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I INTRODUCTION

This document represents the tenth monthly report covering the work on the experimental program for development of an "Osmotic Still" and improvements in the performance characteristics of the Ionics Dual Membrane Fuel Cell during the month of April 1963. This development work is being accomplished under NASA-Lewis Research Center Contract No. NAS 3-2551 by the New Product Research Department of TAPCO, and Ionics Inc. as a subcontractor to TAPCO.

II OVERALL PROGRESS

A. Tapco Portion of Program

1. The design of the 2 KW "Osmotic Still" and the detail drawings have been completed. (Assembly drawing 512-039724-08-1 attached)
2. All of the raw material and parts have been ordered for the still and the rig, however, some of these materials and parts will not be received until the middle of May.
3. Fabrication of the test unit was initiated and nearly completed during this reporting period.
4. The test rig design has been finalized and rig construction is now proceeding.
5. Flaw distribution tests on the electrolyte passage have been completed to determine the internal configuration of the acid cavity to assure uniform distribution over the entire membrane surface. These tests were performed by flow observation through plexiglass end plates. Flow patterns for various internal configurations (baffles, diffusers, etc.) were observed by the injection of colored dye into the flow stream at various locations in the acid cavity. These tests were run at room temperature with water and were qualitative in nature. The configuration selected for good distribution is shown in Figure TRW-1-A.

B. Ionics Portion of Program

1. Three new 5-Cell Batteries were assembled. Four runs of 100 hours or more were completed. In only one of these runs was the voltage not maintained at 3.4 volts or higher at the design current density of 24 amps/ft².
2. The best battery, Battery 3, produced an average voltage of 4.00 volts at 24 amps/ft² over a 133 hour run.
3. A polarization curve taken on Battery 3 indicated an average cell conductance of 369 mhos/ft² and a maximum power density of 64.8 watts/ft² for an average cell.
4. A temperature cycle and leak tests were performed on Battery 4 and a leak test was performed on Battery 3. No leaks were observed on Battery 4 either before or after the temperature cycle and

Battery 3 had, at most, a leak rate less than 0.05% of the gas consumption rate. The temperature cycle appeared to have a small effect on the electrical characteristics of the cell.

5. Components for the 10-Cell Battery were selected.

III CURRENT PROBLEMS

A. Tapco Portion of Program

There have been no major technical problems during this reporting period.

B. Ionics Portion of Program

1. Finalize evaluation of 5 and 10-Cell Battery operation.

IV NEXT MONTH'S EFFORTS

A. Tapco Portion of Program

1. Completion of test rig.
2. Calibration and adjustment instrumentation.
3. Complete Still fabrication.
4. Complete gasket cutting and attachment to Still parts.
5. Assemble Still and install in test rig.

B. Ionics Portion of Program

1. Run 100-hour test on 10-cell battery.
2. Evaluate 5 and 10-cell batteries.
3. Work on design of 2KW unit.

V TEST RESULTS

A. Tapco Portion of Program

1. No test data was recorded during this reporting period. The primary work efforts were devoted to fabrication of the component parts of the 2KW "Still" and assembly of the test stand.

B. Ionics Portion of Program

1.0 Summary of 5-Cell Battery Tests

The data from all of the 5-cell battery tests, including the test results from Batteries 1 and 2 reported last month, have been summarized in three tables. Table I describes the components of the batteries; Table II presents the operating conditions during the runs. In

Table III, the performance of the batteries is shown. Detailed data are presented in Figs. 1 - 27.

In the text following, the performance of each battery is discussed separately.

1.1 Humidification

In the past, the question has been raised whether introduction of very dry gas to the cells might not cause some malfunction due to drying out the membrane and the electrode at the top of the gas compartment. To investigate this possibility, a number of runs were made with the incoming gases "humidified". The gases were passed through flasks containing 3N sulfuric acid at the temperature of the cell. Sufficient disengaging space was available to prevent spray from being carried into the cell. Runs in which the gas was humidified are indicated in Table II:

2.0 Performance of Battery 2

Following run 2A, described in the eighth monthly report, the temperature on Battery 2 was increased to 60°C. The electrolyte rate was reduced to between 5 to 10 ml/min causing the electrolyte pressure to increase to between 4.5 to 5.5 psig. The gases were humidified. These conditions were maintained for 100 hours.

Finally the cell temperature was dropped to 30°C, the electrolyte flow rate was increased to 27 ml/min, the gas pressures were raised to 5 psig to eliminate the pressure imbalance and the humidifiers were removed from the gas feed system. These conditions were maintained for another 16 hours. This run was finally terminated to make space for Battery 5.

The average voltage under load produced by the cell was 30 millivolts higher in run 2B than the voltage maintained in run 2A. This may be due to the higher bath temperatures or the lower electrolyte rate (i.e., higher internal temperature). However, in contrast to the relatively small and random variation of voltage in 2A, the voltage in 2B drops steadily from a value near 3.9 to just below 3.4 volts at the end of the run.

The voltage in the recapitulation run, 2C, is 100 millivolts below that in 2A. Over the 16 hours that the cell was run at 30°C, the voltage rose slowly, and given time, might have reached the value observed in run 2A.

There is a pronounced increase in liquid accumulation on both sides of the battery in the 60°C run. It will be noticed in further discussion of 5-cell batteries that, when the same battery is run at two different temperatures, the liquid accumulation is consistently higher in the higher temperature run. This effect is more pronounced on the hydrogen side. This liquid production might provide some explanation for the decline in the voltage during run 2B.

The gas pressure drops in run 2B are similar to, but slightly higher than, those measured in runs 2A and 2C.

2.1 Performance of Battery 3

Except for Niobium pusher and collector plates, Battery 3 is identical in construction to Battery 1.

This battery was first operated for 133 hours at 60°C (Run 3A). After this a polarization curve was taken, the cell was lead-tested, and a run at 30°C was begun (Run 3B). After 51 hours of operation, a cell failure forced the termination of the run.

Two system failures occurred during these runs. In Run 3A, the hydrogen humidifier upset, sending 3N sulfuric acid through the hydrogen compartments. The cell recovered from this accident rapidly giving 0.1 volt increases in voltage for a short time after the gas flow was restored. Run 3B was terminated by a fire in the battery after a failure of the electrolyte circulating system. The fire caused holes in several of the membranes.

The voltage under load of this battery averaged the very high value of 4 volts during run #a. The voltage showed a slow decline from almost 4.3 volts to 3.84 volts after 133 hours. The polarization curve, Figure 1, measured directly after this run showed the remarkably high specific conductance of 73.7 mhos/ft². This equals an average of 369 mhos/ft² for a single cell which is considerably higher than was ever measured for a single cell before. A power density curve is presented in Figure 2.

Run 3B, at 30°C, produced a lower, but relatively stable, voltage centering about 3.74 volts.

For a leak test, the cell was submerged in water in the constant temperature bath and carefully observed for leaks. Approximately 2cc/hr of gas was seen which could not be clearly attributed to electrolysis of the bath water caused by the 4 volts existing between the terminal collector plates.

On the oxygen side, liquid accumulations were almost equal for the two runs. However, the hydrogen side accumulation of both acid and water in the 30°C run was one tenth that of the 60°C run.

The gas pressure drops were, on the average, higher than in previous cells. The hydrogen side pressure drop in run 3B is probably abnormally low due to the very small quantity of liquid which had to be pushed out of the compartment. A very marked trend is seen in run 3A where the oxygen pressure drop increases from 75 to 140 mm water and the hydrogen from 95 to 140 mm water over the course of the run. No such relationship exists in run 3B.

2.2 Performance of Battery 4

This battery uses penton compartments and dynel-backed membranes. Battery 4 was first run for 134 hours in a 60°C bath. A polarization curve was recorded and the battery was leak-tested. This run was arbitrarily broken into three sub-parts in presenting the data in order that a very interesting bit of behavior in the liquid accumulation might not be obscured. This is discussed below. The battery was then run for 17 hours at 30°C enough to reach steady-state operation. Then the bath temperature was raised to 60°C, the leak-test was repeated and another polarization curve was recorded. The cell was then run again at 60°C for 26 hours. This test was terminated when some of the components were needed for the assembly of another battery.

The average voltage for the 134 hour run was 3.5 volts, lower by almost 100 millivolts than Batteries 1 or 2 under similar conditions. Only for a brief period after 100 hours did the voltage drop below 3.4 volts. However, when the temperature was restored to 60°C, only 3.339 volts were recorded.

Two polarization curves were recorded before and after the 30°C run. These indicate that some significant change occurred in the cell. The first shows a specific conductance of 33.3 mhos/ft², the second an increase to 43.5 mhos/ft², indicating that above that current the cell operated better after temperature cycling. The significance of this behavior lies most likely in the position of the interface, which has not been investigated sufficiently to warrant any interpretation.

The leak test consisted, again, of submerging the cell completely in the bath. In both leak tests, a current of 20 amperes reduced the potential between the terminal end plates below that necessary for electrolysis, under which conditions no bubbles were observed rising from the cell.

The significant part of the liquid accumulation data is the sulfuric acid rate through the hydrogen side membrane. During period 4-A-2, the sulfuric acid rate dropped from 0.32 (not large by comparison to other cells) to 0.04 grams/hr. This appears to be a result of the electrolyte normality change. Normally the normality drops slowly as a result of the absorption of product water. When the normality dropped below 4N, the flow of acid through the membrane dropped sharply. Upon addition of concentrated sulfuric acid to the reservoir, made periodically to bring up the concentration, the flow returned to its former value. In corroboration, the water flow shows a simultaneous but fractionally smaller decrease.

The liquid accumulation rates for run 4C are not in line with those of 4A but it is possible that they are influenced by run 4B and by the taking of the polarization curves.

The gas pressure drops in run 4A average higher than those of run 3. In run 4B, they are almost identical to those of run 2C. But there is a considerable increase, particularly on the hydrogen side in run 4C. Upon disassembly, it was found that the gaskets had swelled over the gas inlet channels, probably as a result of heat generated within the cell during the taking of polarization curves, to the point where these channels appeared to be almost shut off.

2.3 Performance of Battery 5

Battery 5 was similar in construction to Battery 2 except for the use of Zirconium pusher and collector plates and dacron-backed membranes. This battery was intended to have gas and liquid compartments of Halon. However, deliveries of this material in satisfactory thicknesses was so slow that Teflon components were substituted. Thus, Halon compartments were not evaluated.

The first assembly of Battery 5 operated below 3.4 volts from the very beginning at 30°C, run 5-A-1. To improve the performance, the bath was heated to 60°C. At this temperature, the cell voltage dropped from a starting high of 3.47 volts to 3.38 volts in 18-1/2 hours. Consequently, the cell was disassembled and two changes were made. The electrodes were shuffled and the oxygen electrodes which had shown the poorest performance were replaced. It has been noticed that the oxygen electrode in a cell exerts considerably more influence over the performance than the hydrogen electrode. Consequently, unsatisfactory oxygen electrodes can be used on the hydrogen side with little bad effect. It was also observed that the Zirconium collector plates, because of the relatively higher resistance of Zirconium, were lowering the voltage of the first and last cells of the battery by about 30 millivolts each. Since the current path in the terminal plate, the resistance there is more important; furthermore, considerable electrolysis had occurred along the edges of the collector plates accompanied by noticeable removal of metal. To avoid both of these difficulties, terminal collector, and pusher plates of tantalum were substituted. After these adjustments, run 5B was made, lasting for 117 hours. This run was terminated when the need arose for components for the 10-cell battery.

The electrical performance of Battery 5 was only marginal. While no measurements taken with the calibrated potentiometer were poorer than the criterion of 3.4 volts at 6 amperes, the recorder, a less reliable instrument, showed several points below 3.4 volts. On two occasions surges of electrolyte and gas were used to improve the performance of the battery.

A good comparison of the effect of temperature on liquid accumulation is shown by Battery 5. The runs at 30°C, 5-A-1 and 5B give very similar rates, which are significantly lower than those observed at 60°C, run 5-A-2. On the hydrogen side, both water and acid rates

are 2 - 3 times as high in the 60°C run.

The pressure drops measured in run 5B averaged slightly higher on the hydrogen side and considerably lower (66 vs. 100) on the oxygen side when compared to run 2A.

3.0 Overall Conclusions From 5-Cell Battery Runs

Niobium is the most desirable pusher and collector plate material of those investigated.

The voltages produced by the cell using niobium are, on average, higher than any other cell has produced. This is not thought to be due to the internal resistance of the metal but to the surface resistance which is obtained under the conditions of the cell. Note the exceptionally high specific conductance of the cell measured in run 3A.

Either Taflon or Penton can be used for gas and electrolyte compartments. No evidence of deterioration of either material during normal testing was observed.

Gas humidity seems to have an effect on the electrical output of the cell at 60°C.

In the three humidified runs at 60°C, 2B, 3A and 4A, a noticeable decrease of voltage with time is present. This behavior is not seen in the 30°C humidified runs, 3B and 5B, or in the runs where the gas was not humidified, 1A and 2A.

Dynel-backed membranes are inferior to glass-backed membranes. No direct comparison can be made since no two batteries differed by only the type of membrane. However, the two poorest batteries both contained Dynel-backed membranes.

More liquid is carried out in the gas streams in the 60°C runs than in the 30°C runs.

This liquid transport is not entirely due to non-selective leakage since the concentration of the liquid is, in general, not equal to the electrolyte concentration. Further, either the concentration of material transported is temperature sensitive or else two transport processes occur simultaneously, their relative rates depending on temperature, since the concentration of the liquid in the gas stream are not the same at 60°C as at 30°C.

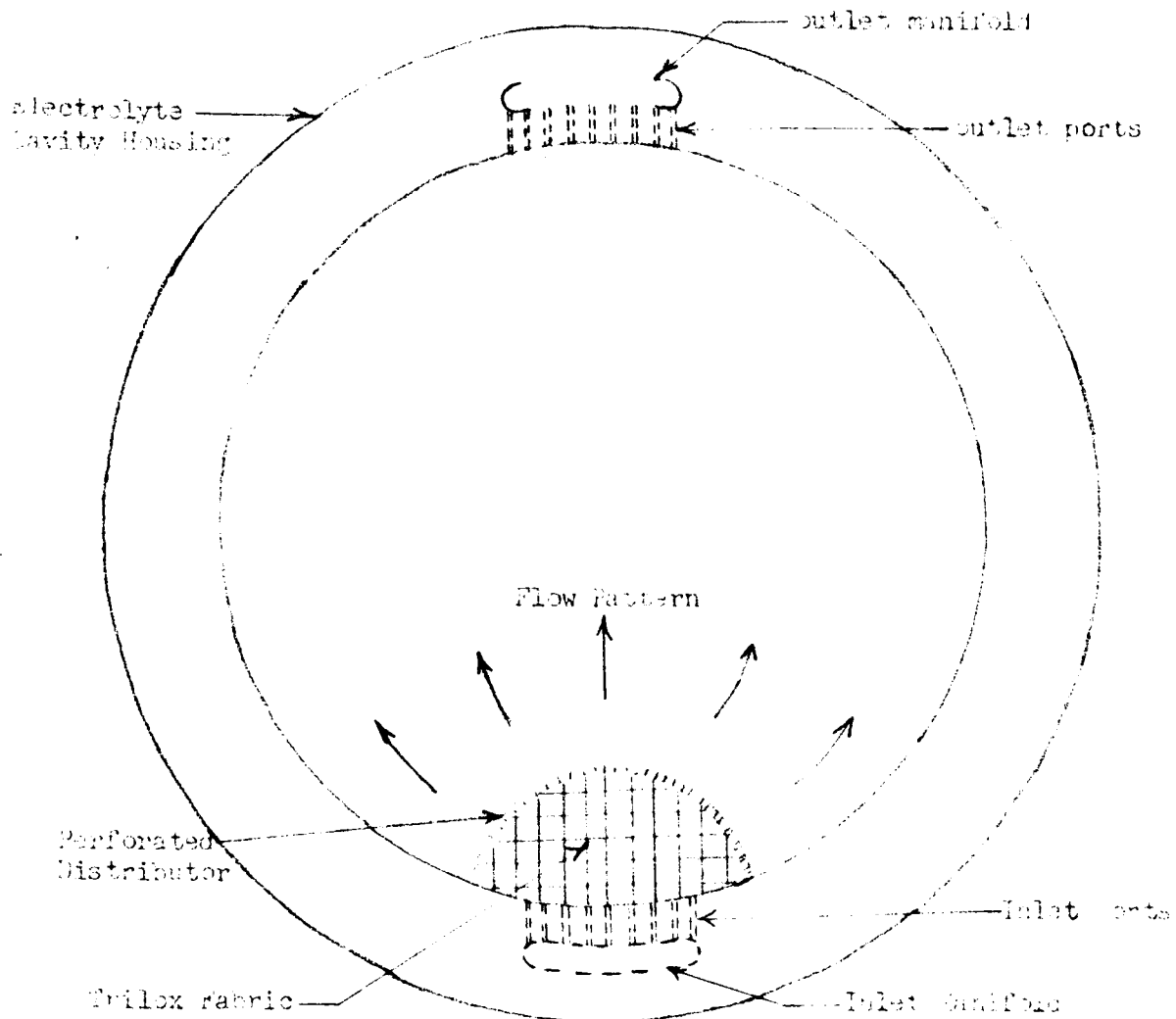
For some runs, the quantity of liquid appearing in the gas stream was quite small. See runs 2-A-2 and 3B (hydrogen side). The reason for the variation in liquid accumulation rates is not currently known.

Pressure drops of about 25 mm of water appear to be adequate for distribution of gases.

While there have been cases of single cells in a battery which performed notably poorer than the others, except for run 1B, where the flow rates were very small and the pressure drops were only 15 mm of water, there has been no example of one cell showing the precipitous drop which occurs when gas flow is shut off while the rest of the cells in the battery continued to operate.

FIGURE

ELECTROLYTE FLOW DISTRIBUTION ARRANGEMENT



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TABLE I

DESCRIPTION OF 5-CELL BATTERY COMPONENTS

COMPONENT	BATTERY 1	BATTERY 2	BATTERY 3	BATTERY 4	BATTERY 5*
End Plates	-----	3/8" Stainless Steel	-----	-----	-----
Insulators	-----	60 mil Butyl Rubber	-----	-----	-----
Pusher & Collector Plates	10 Mil Tantalum	10 Mil Tantalum	10 Mil Niobium	10 Mil Tantalum	10 Mil Zirconium
Electrodes	-----	Standard Sintered Teflon	-----	-----	-----
Membranes	61 AZG**	61 AZG	61 AZG	61 AZL***	61 AZL
Gas Compartments	64-65 Mil Teflon	64-65 Mil Teflon	64-65 Mil Teflon	60-65 Mil Penton	64-65 Mil Teflon
Electrolyte Compartments	118-133 Mil Teflon	99-102 Mil Teflon	118-133 Mil Teflon	110-115 Mil Penton	99-102 Mil Teflon
Trilok Filler	-----	Type 6027-1-1	-----	-----	-----
	2 pc/cell	1 pc/cell	2 pc/cell	2 pc/cell	2 pc/cell
Gaskets & Grommets	-----	8 Mil Dacron-Backed Viton	-----	-----	-----

* In Run 5B, Tantalum Terminal Collector and Pusher Plates were used (see Text)

** IONICS 8 oz. Glass-Backed Membrane

*** IONICS Dynel-Backed Membrane

Four Grommets used for each Collector Plate

TABLE II

OPERATING CONDITIONS OF 5-CELL BATTERY TESTS

RUN NUMBER	1A	1B	1C	2A1	2A2	2B	2C	3A	3B	4A1	4A2	4A3	4B	4C	5A1	5A2	5B
<u>Feed Rates</u>																	
H ₂ liters/min	2.1	0.55	0.90	2.2	2.2	2.1	2.0	2.1	2.1	2.2	2.1	2.0	2.1	2.0	1.2	2.1	2.2
O ₂ liters/min	1.02	0.21	0.44	1.05	1.05	1.0	1.1	1.1	1.1	1.0	1.0	1.0	1.2	1.0	0.8	0.65	1.0
Electrolyte ml/min	60	7	7	60	60	8	27	24	10	34	36	13	12	8	30	10	11
<u>Pressures</u>																	
H ₂ , psig.	5.0	5.0	5.0	2.5	3.0	3.1	5.4	5.1	5.2	5.2	5.3	5.3	5.5	5.0	4.8	5.2	4.9
O ₂ , psig.	5.0	5.0	5.0	2.1	3.2	3.1	5.2	4.9	4.8	4.7	4.7	5.0	5.0	5.1	5.0	4.5	4.8
Electrolyte, psig.																	
High	5.1	5.1	5.1	5.1	3.0	5.5	5.5	5.2	5.1	5.3	5.1	5.5	5.4	5.4	5.2	5.2	5.4
Low	4.7	4.7	4.7	4.7	3.0	4.5	4.5	4.4	4.5	4.0	4.0	4.0	4.5	4.1	4.5	4.5	4.8
<u>Current Load</u>																	
amps/ft ²	24	24	24	24	24	24	24	24	24	24	24	24	24	24	24	24	24
<u>Temperature °C</u>																	
Duration, hours	60	60	60	30	30	60	30	60	30	60	60	60	30	60	30	60	30
<u>Humidification</u>																	
	112	24	27	48	52	100	16	133	51	49	68	17	17	26	16	20	117
	No	No	No	No	No	Yes	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes

TABLE III

PERFORMANCE DATA FROM 5-CELL BATTERY TESTS

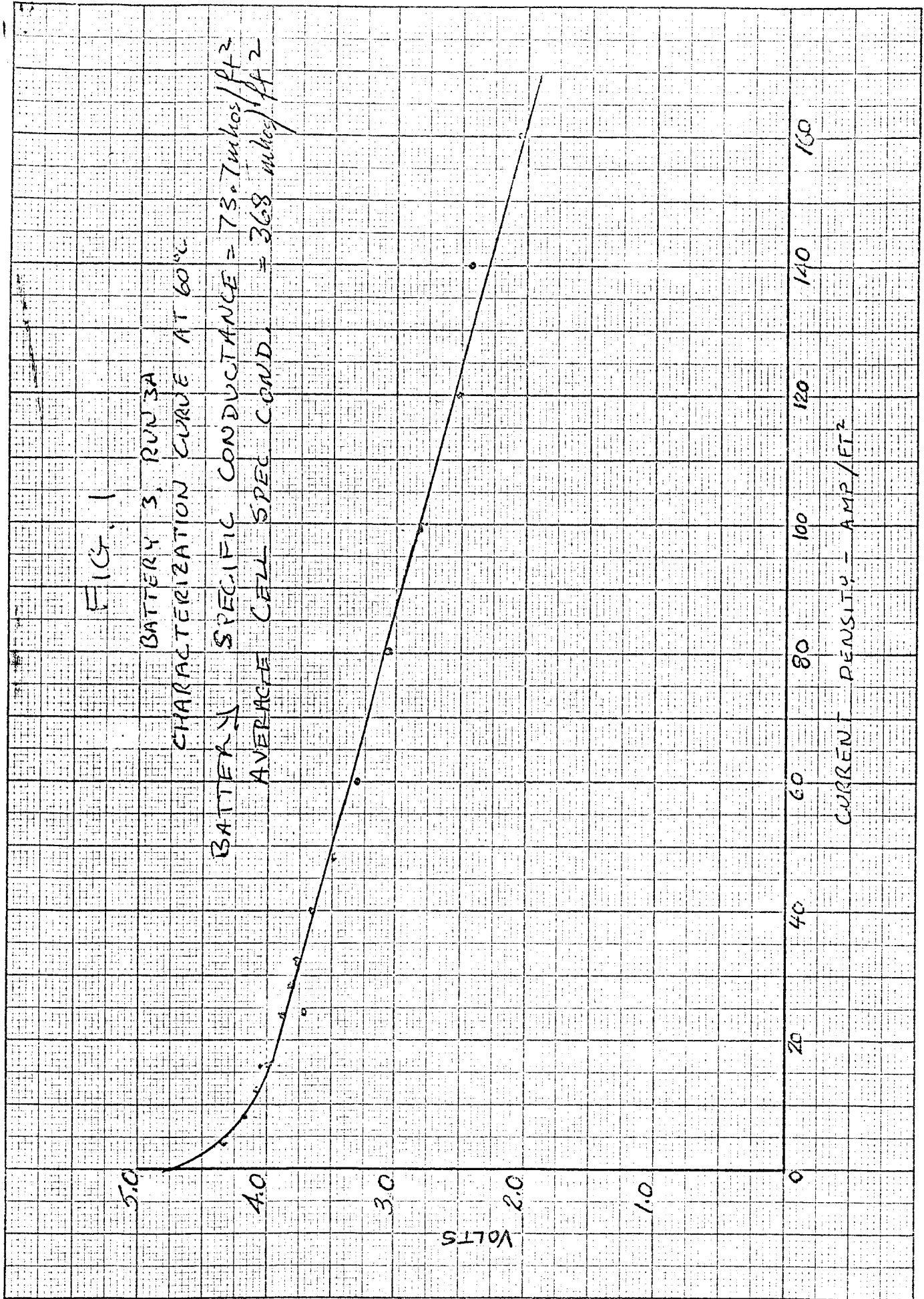
RUN NUMBER:	1A	1B*	1C	2A1	2A2	2B	2C	3A	3B	4A1	4A2	4A3	4B	4C	5A1	5A2	5B
PRESSURE DROP (mm H ₂ O)																	
H ₂	46	15	25	50	50	81	67	125	100	175	123	125	53	280	32	27	58
O ₂	60	14	30	100	100	142	123	115	140	120	215	160	132	172	57	42	66
Electrolyte	175	52	52	-	-	-	-	95	-	-	-	-	-	-	-	-	-
LIQUID ACCUM. (gms/hr)																	
H ₂ Side																	
H ₂ O	6.6	5.5	7.6	4.8	1.35	18.5	5.8	10.6	1.02	12.4	12.4	12.4	-	6.3	2.3	8.27	2.94
H ₂ SO ₄	1.4	1.6	0.95	1.4	0.26	3.9	1.8	1.15	0.09	1.4	1.4	1.4	-	0.76	0.71	1.54	0.64
O ₂ Side																	
H ₂ O	5.2	4.3	7.5	7.5	2.6	18.8	7.1	11.8	10.9	4.8	4.5	4.8	-	11.5	4.46	9.85	7.0
H ₂ SO ₄	1.1	1.1	1.25	1.8	0.47	4.5	1.8	2.0	2.55	0.32	0.04	0.32	-	1.3	1.07	1.5	1.2
VOLTAGE (@ 24 A/ft ²)																	
Cell 1	.670	.684	.684	.670	.678	.680	.648	.790	.728	.723	.692	.712	.710	.702	.614	.632	.695
Cell 2	.745	.760	.753	.795	.785	.742	.728	.820	.765	.714	.684	.694	.694	.661	.722	.727	.720
Cell 3	.715	.728	.726	.670	.690	.717	.704	.804	.764	.719	.688	.718	.698	.633	.701	.708	.704
Cell 4	.700	.718	.716	.790	.770	.764	.722	.802	.747	.710	.676	.681	.670	.653	.716	.721	.710
Cell 5	.730	.740	.726	.658	.650	.712	.672	.789	.744	.737	.686	.700	.691	.690	.627	.640	.682
Total	3.560	3.630	3.605	3.585	3.575	3.615	3.474	4.005	3.748	3.603	3.426	3.505	3.483	3.339	3.380	3.428	3.511
CONDUCTANCE (mhos/ft ²)																	
Cell 1								294				200		223			
Cell 2								415				174		213			
Cell 3								364				200		229			
Cell 4								425				109		223			
Cell 5								375				186		208			
Total								73.7				33.3		43.5			

*Operation unstable, occasional gas surges needed to clear ports.

FIG. 1

BATTERY 3, RUN 3A
CHARACTERIZATION CURVE AT 60°C

BATTERY SPECIFIC CONDUCTANCE = 73.7 mhos/ft^2
AVERAGE CELL SPEC. COND. = 36.8 mhos/ft^2



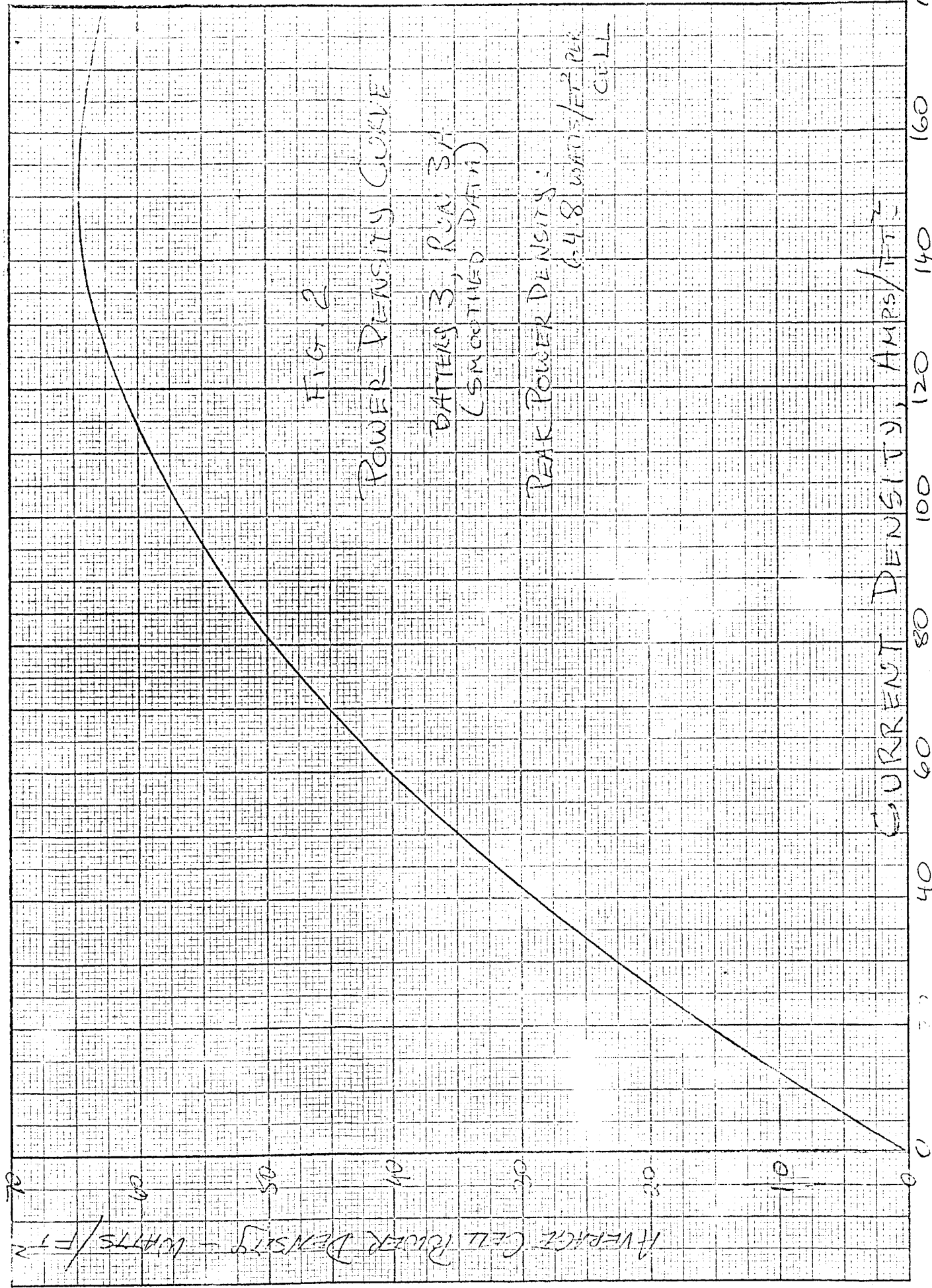


FIG. 3

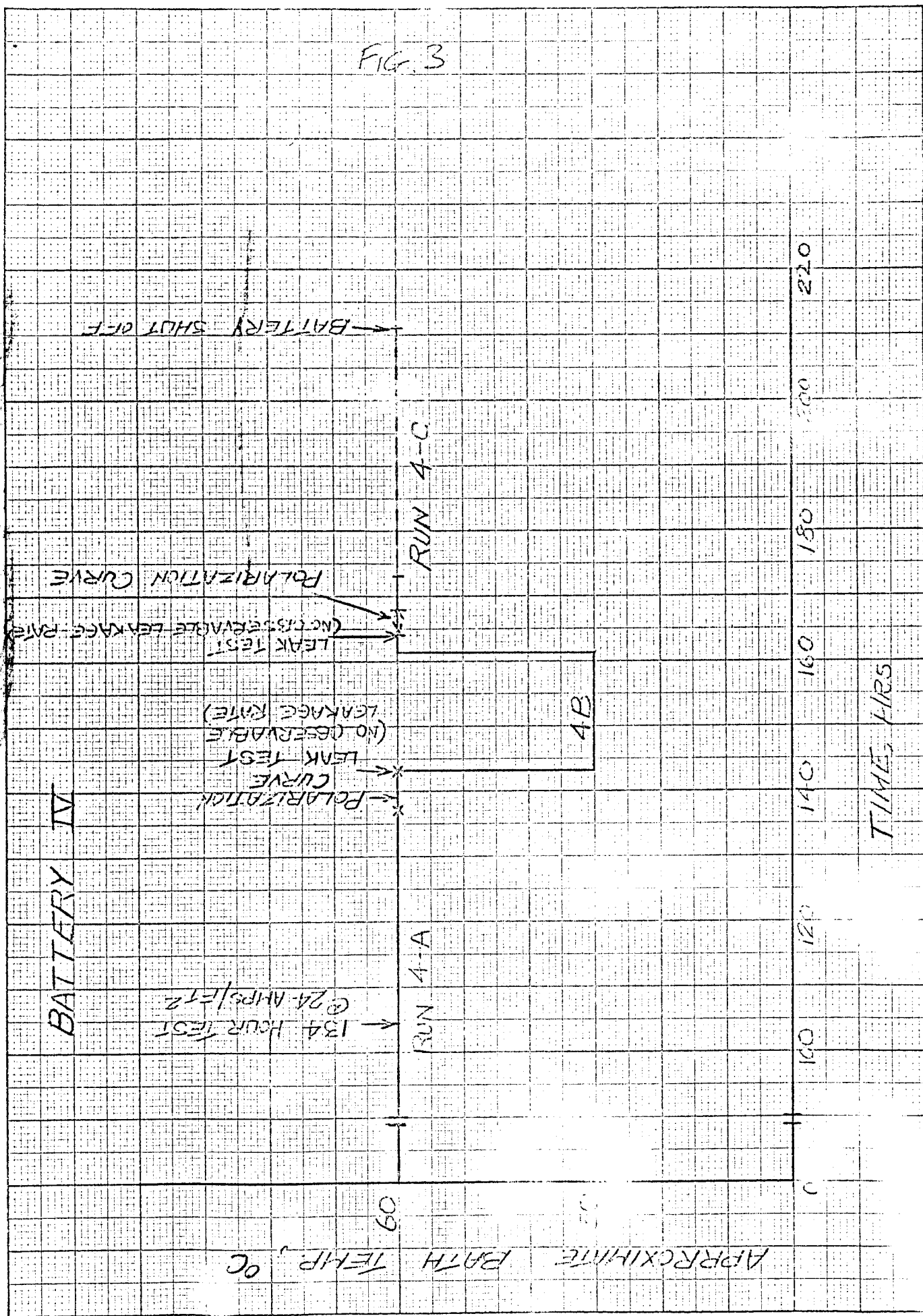


FIG 4

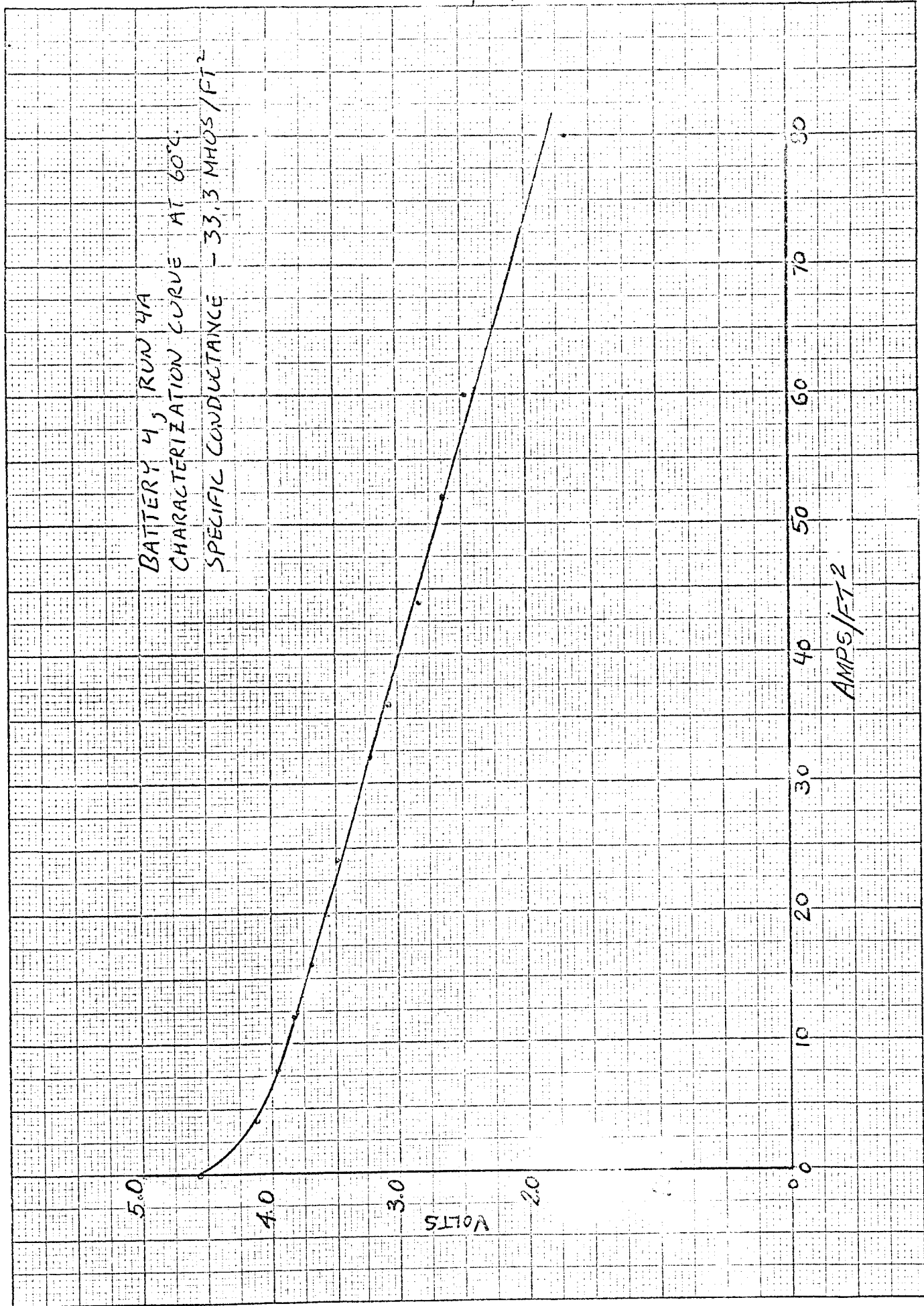


FIG 5

BATTERY 4, RUN 4C
CHARACTERIZATION CURVE AT 60°C
AFTER TEMPERATURE CYCLING
SPECIFIC CONDUCTANCE - 43.5 MHOS/FT²

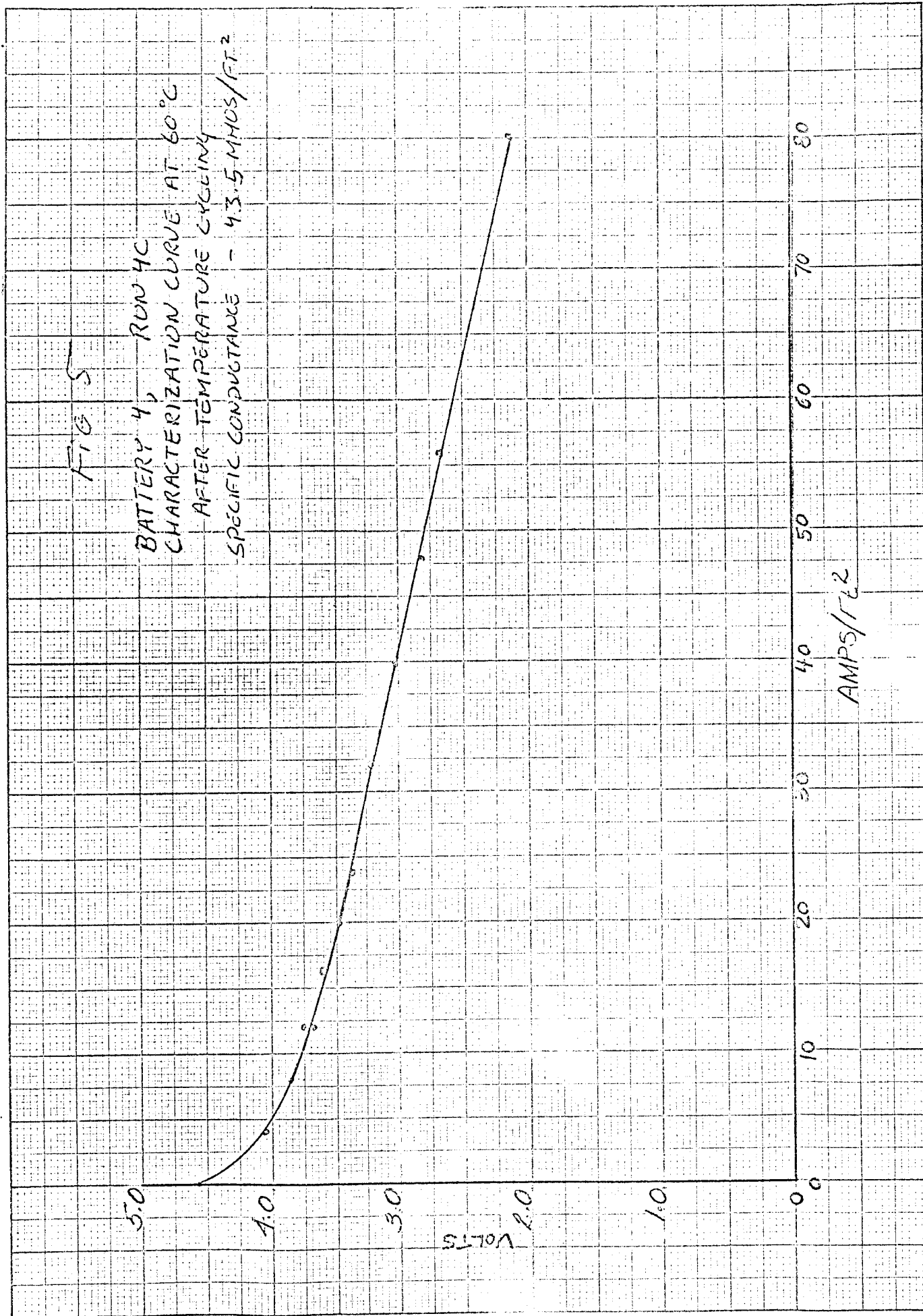


FIGURE 6

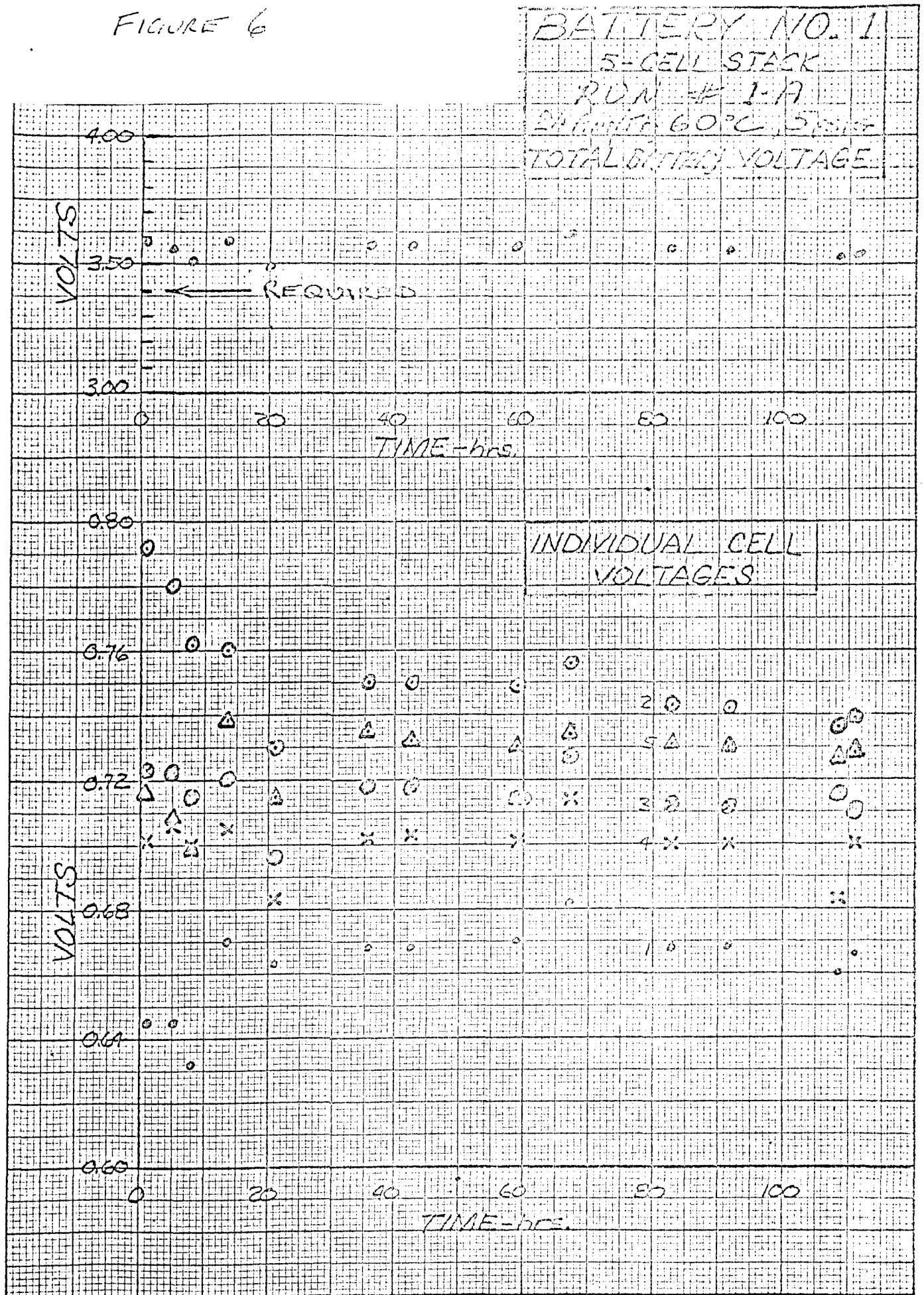


FIGURE 7

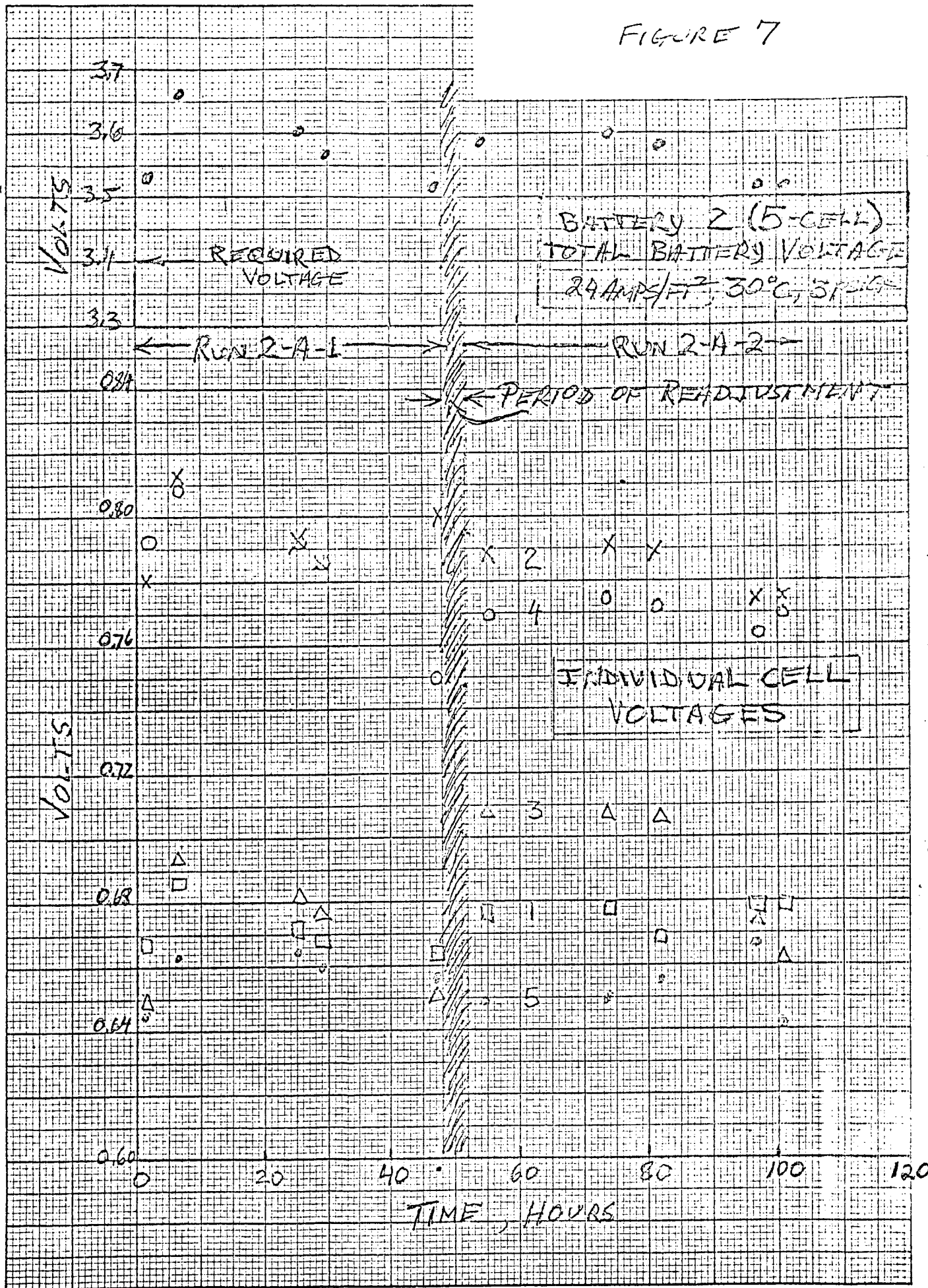


Fig 8

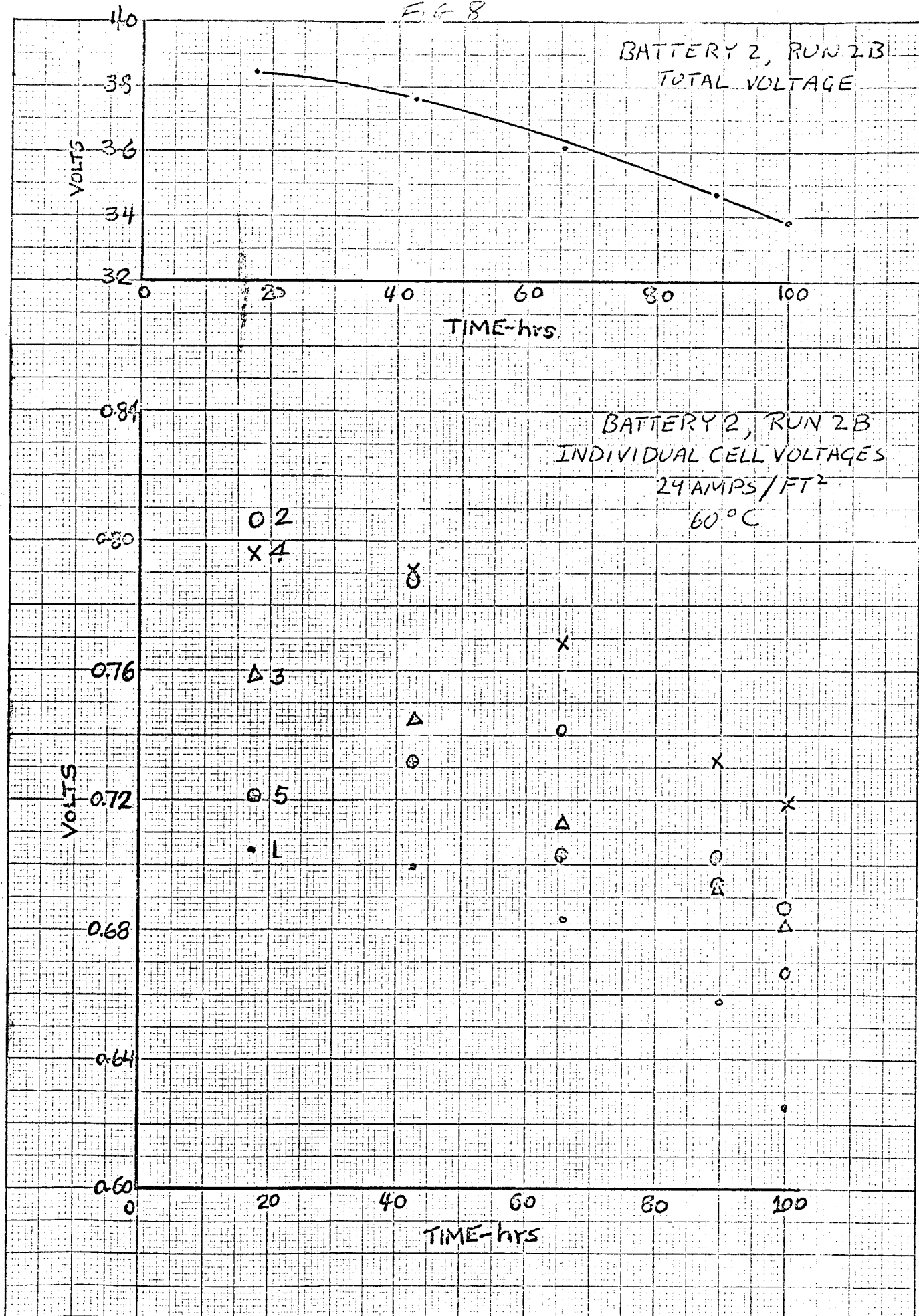
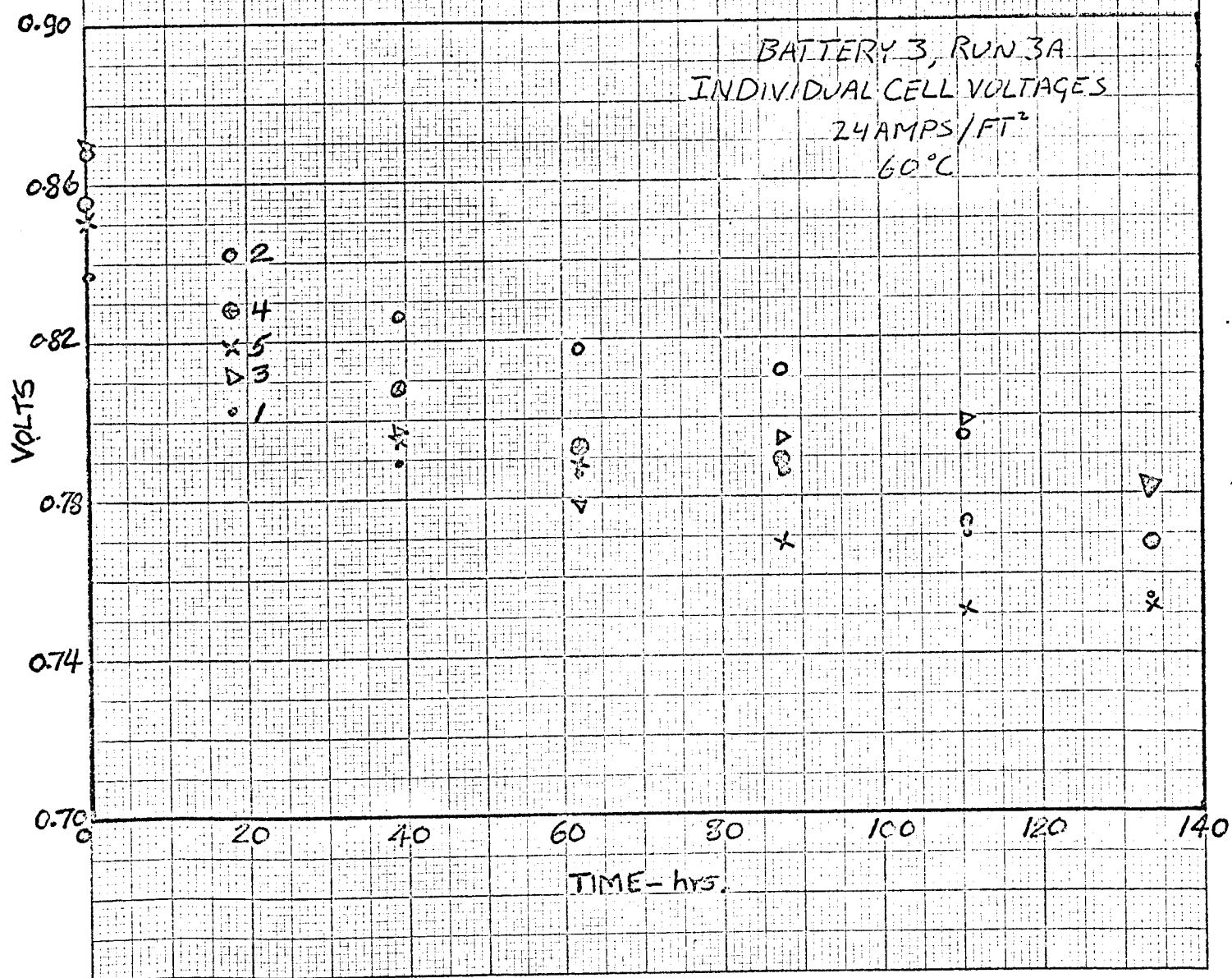
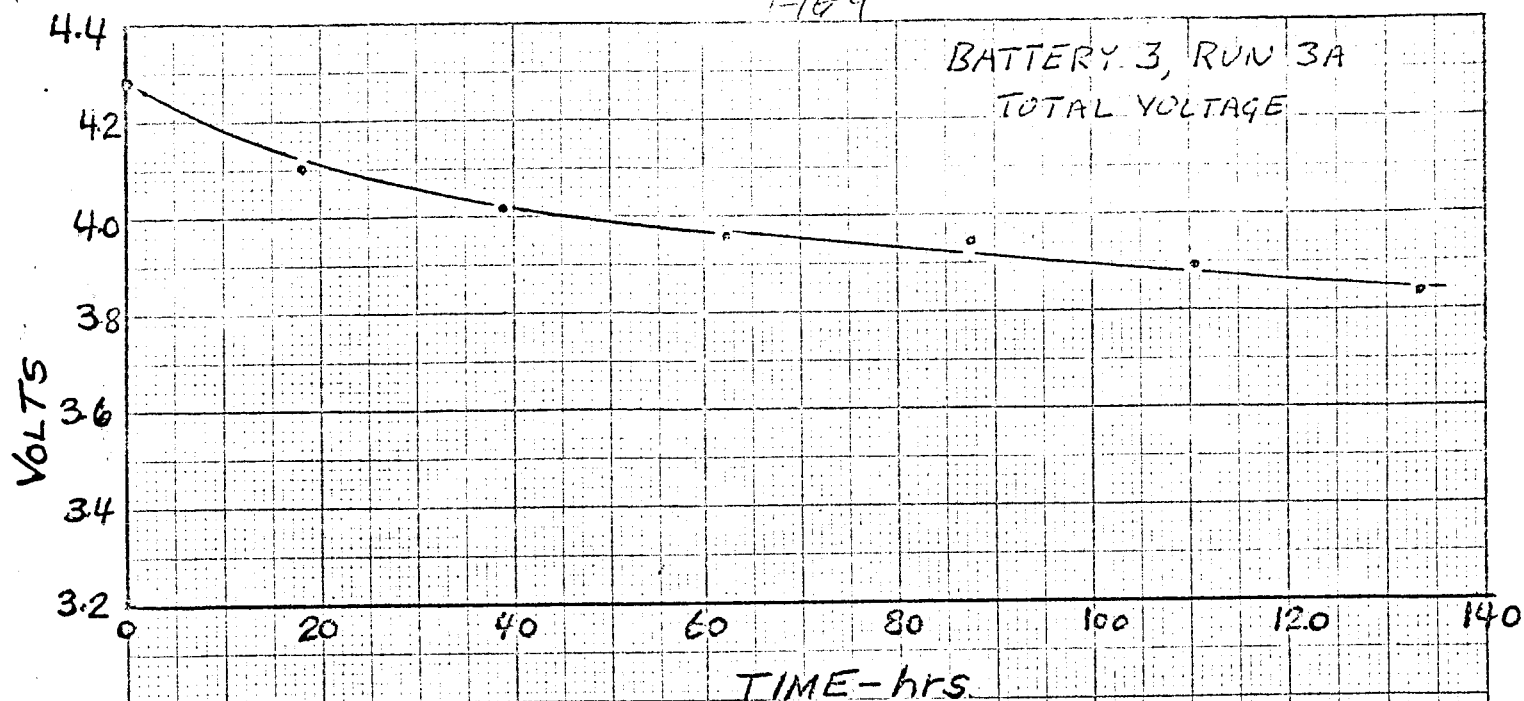


Fig 9



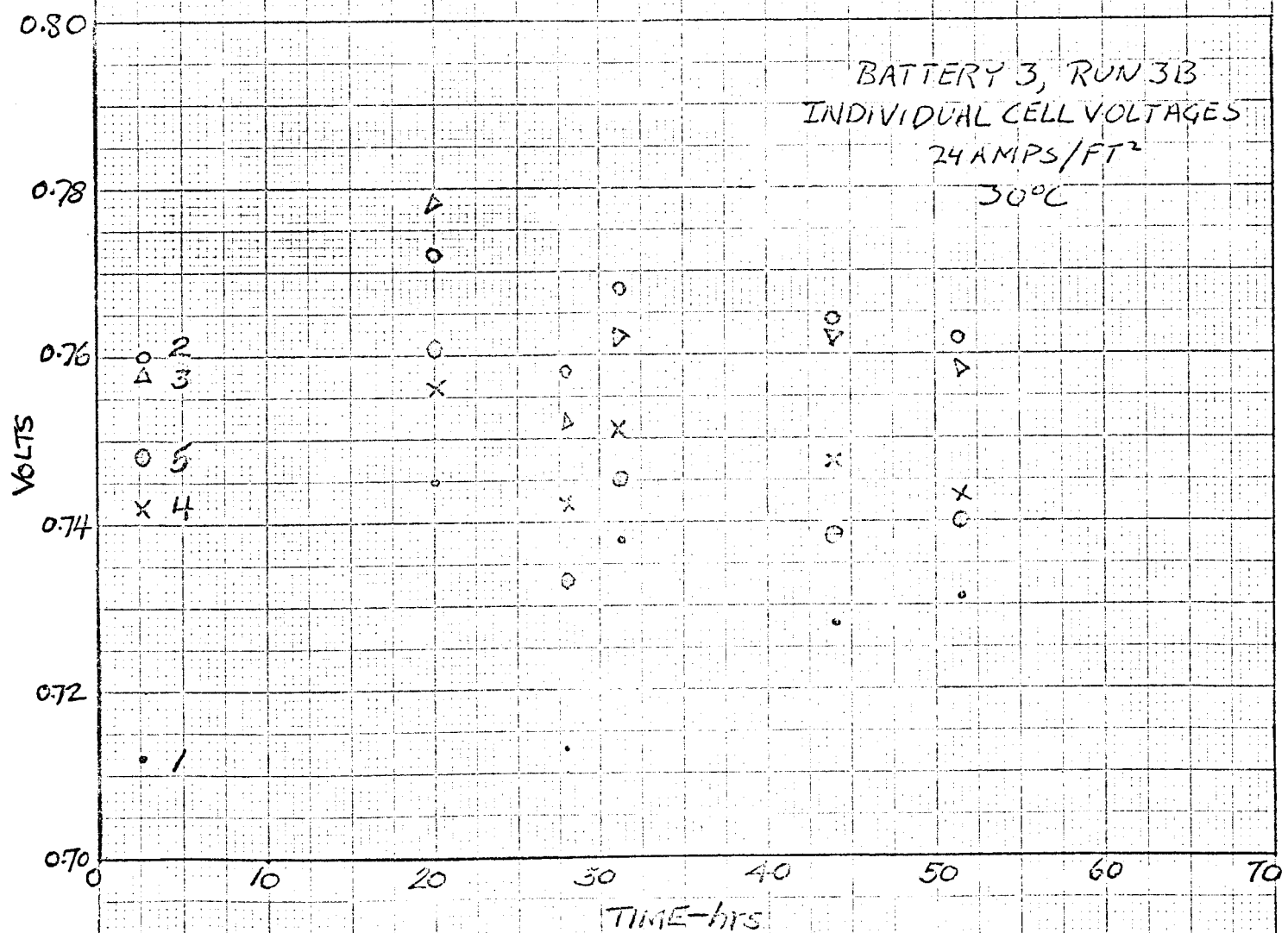
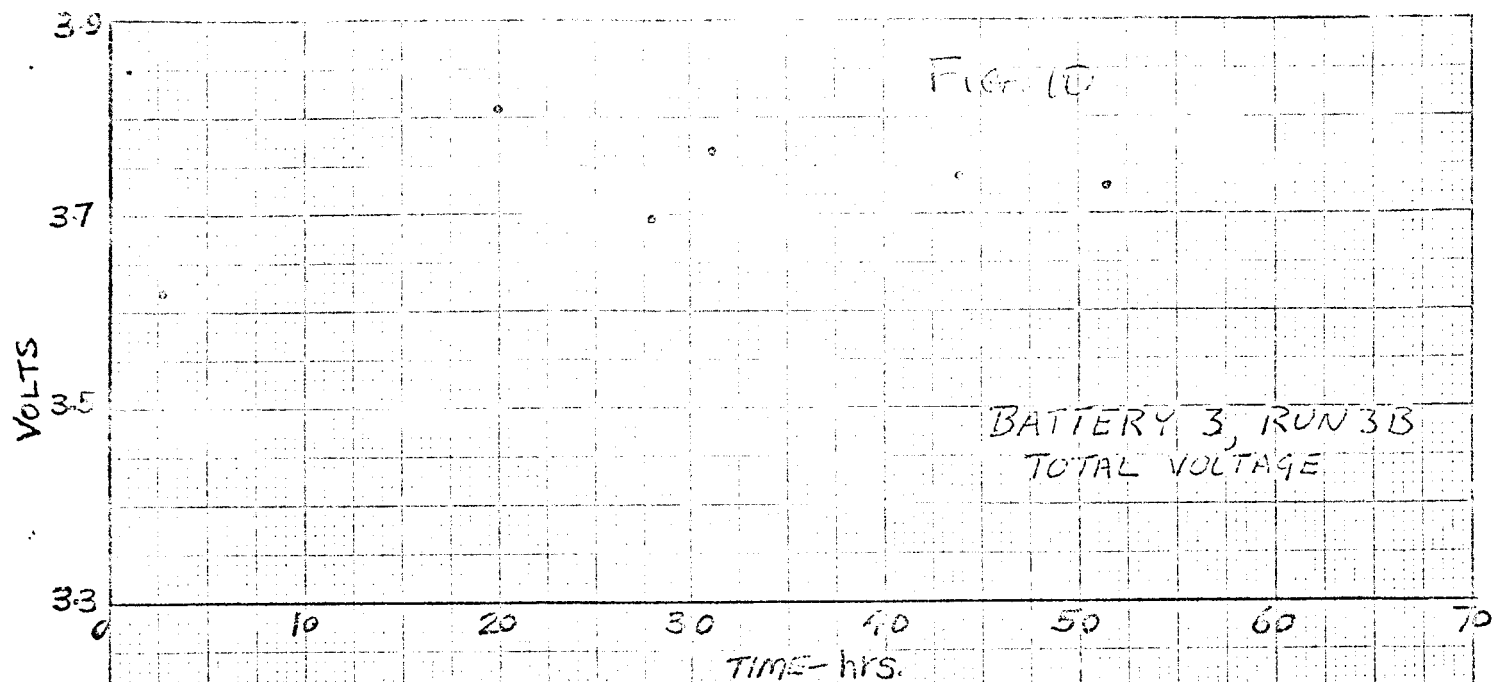
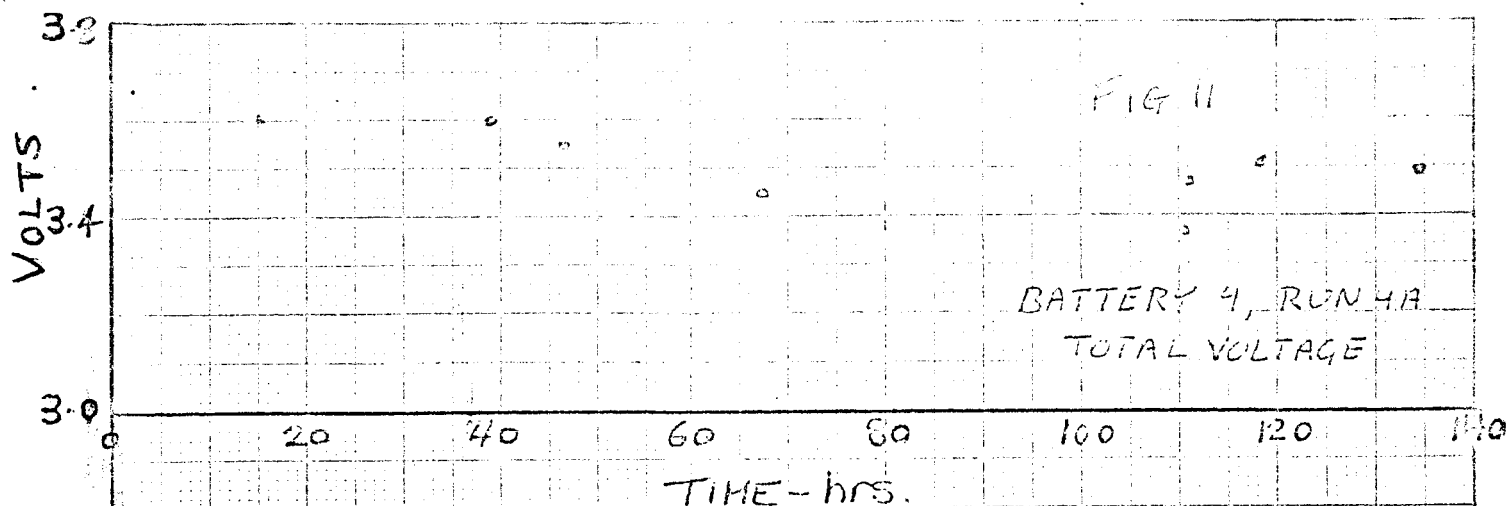
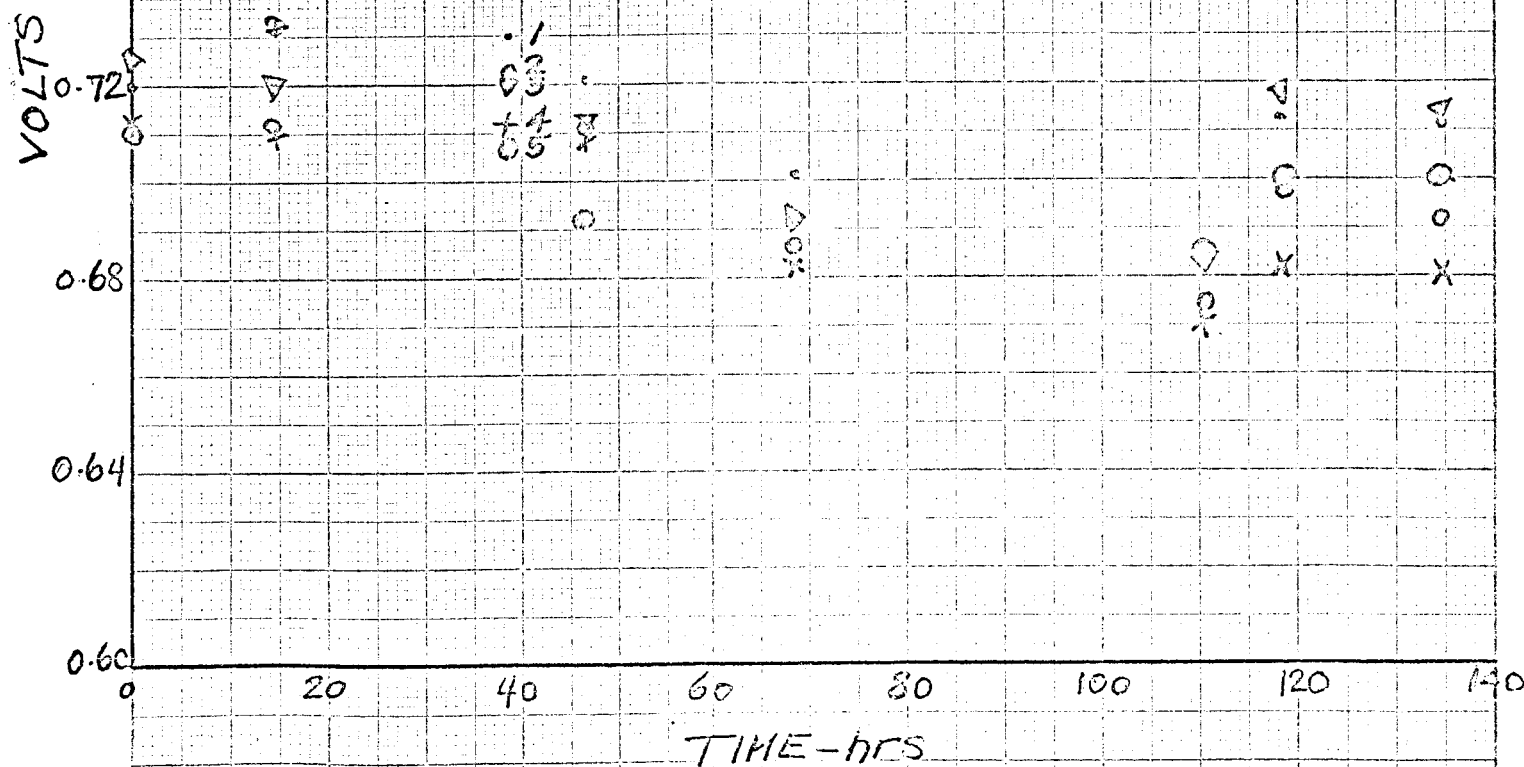


FIG 11

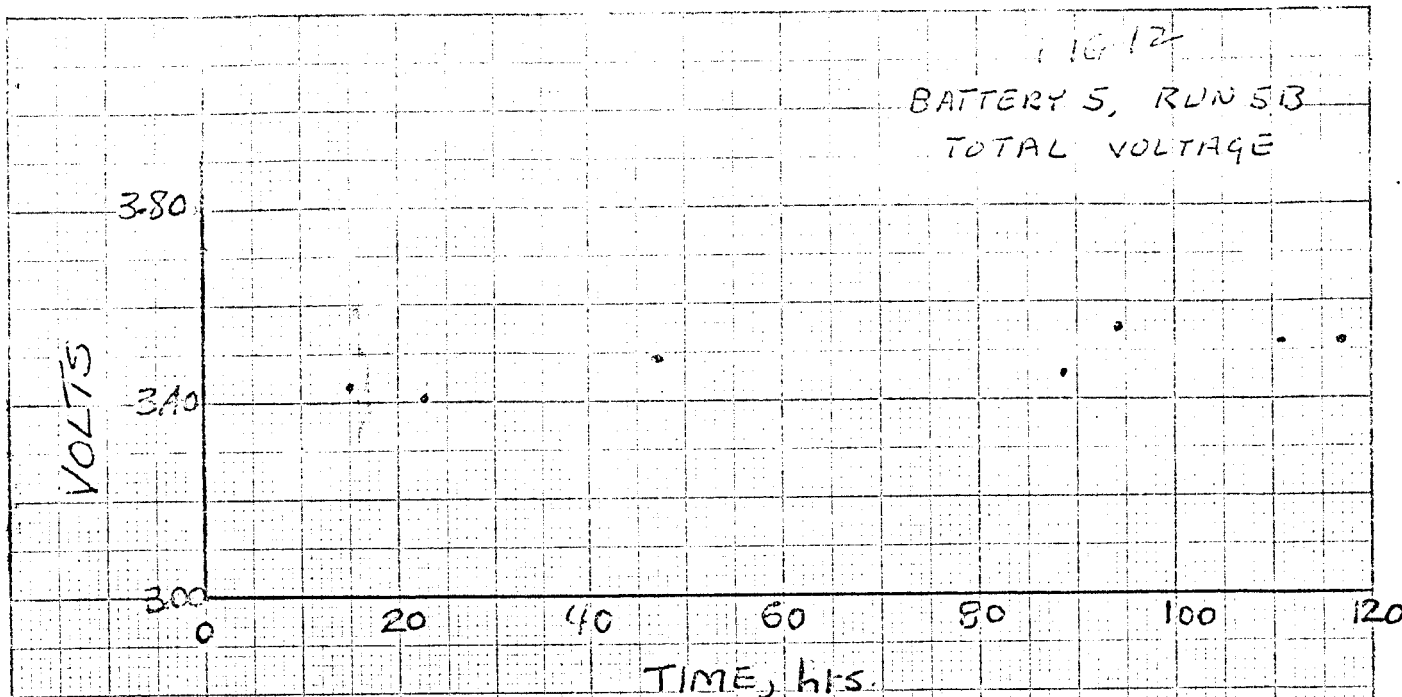
BATTERY 4, RUN 4A
TOTAL VOLTAGE



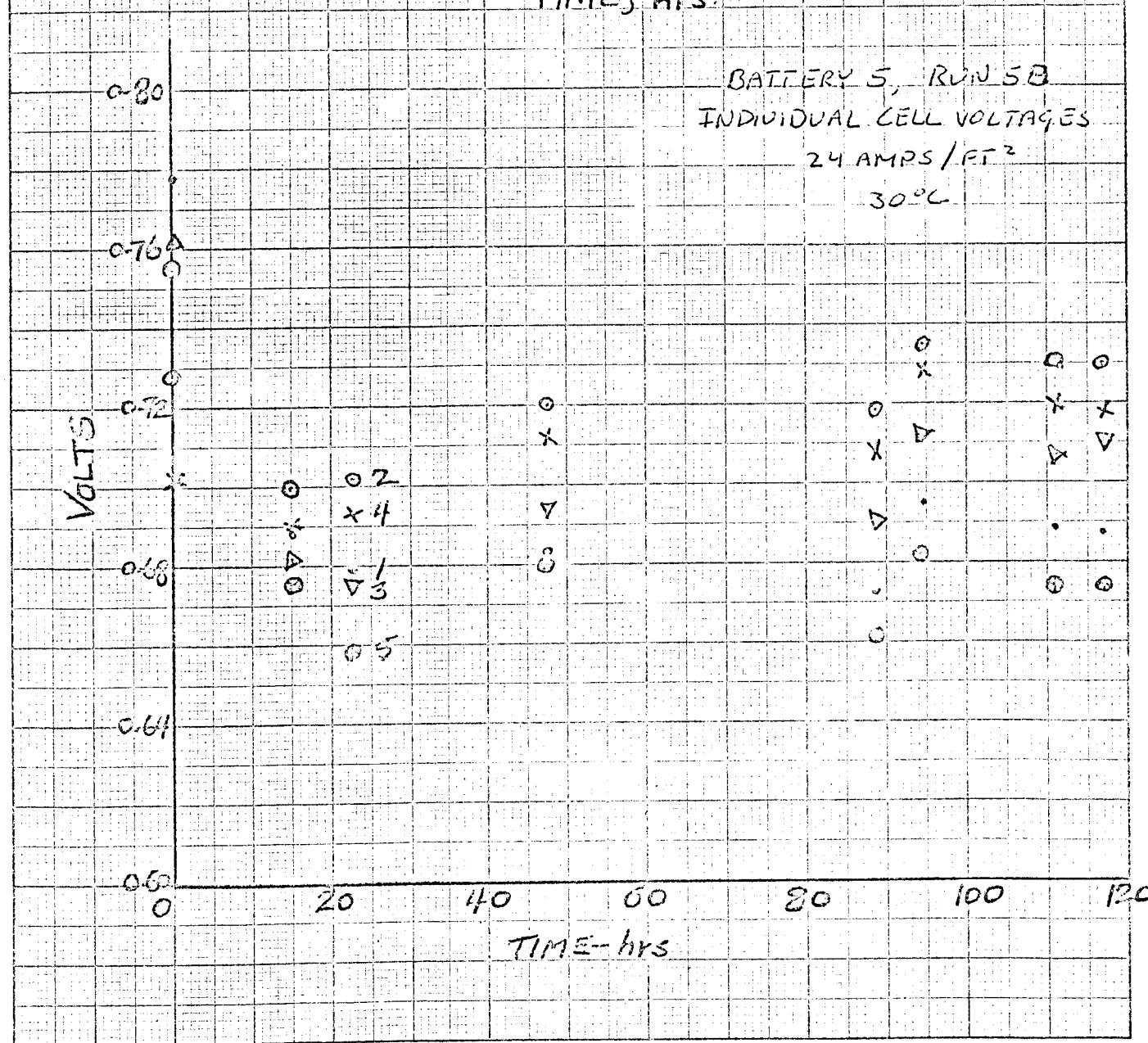
BATTERY 4, RUN 4A
INDIVIDUAL CELL VOLTAGES
24 AMPS / FT²
60°C

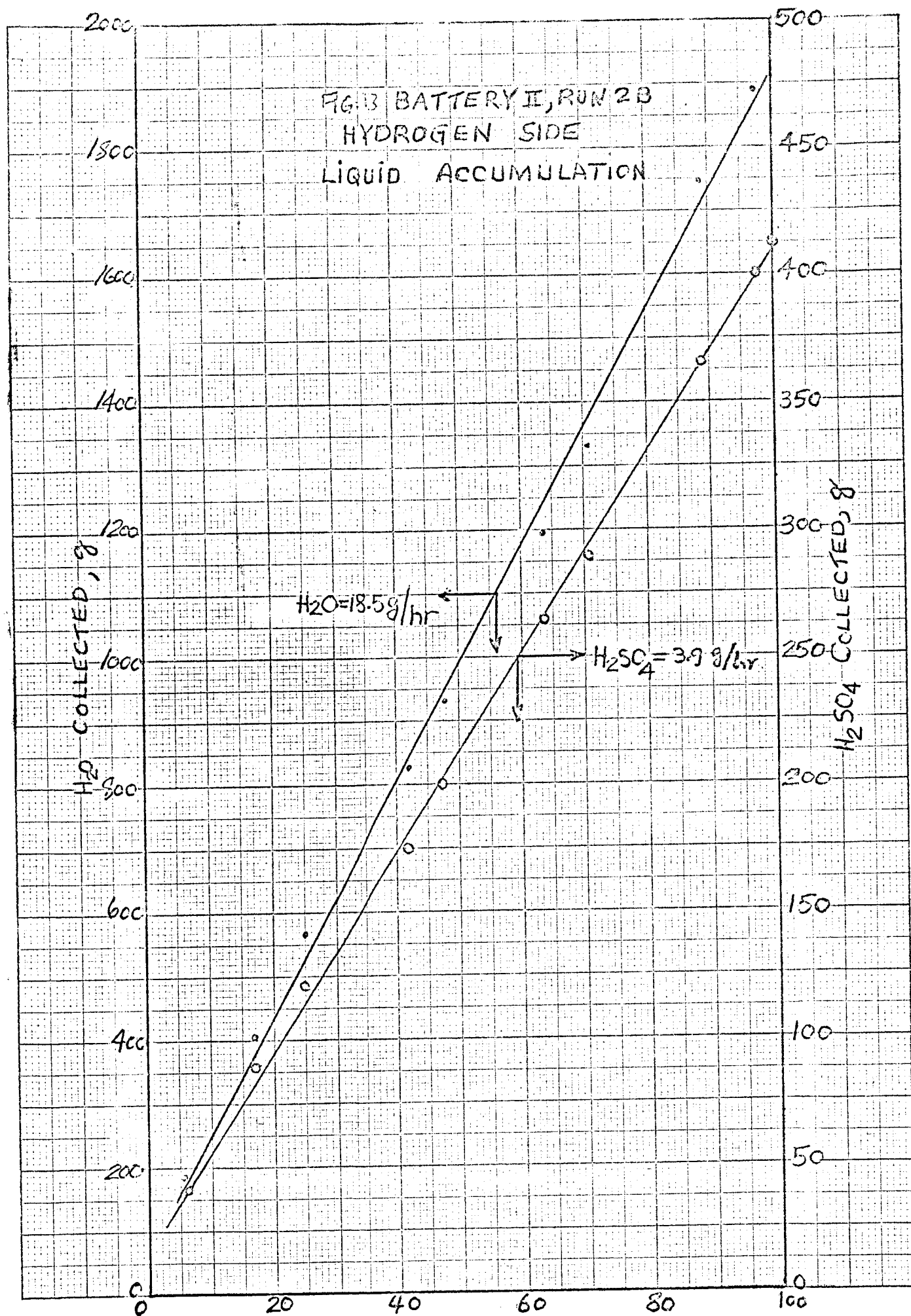


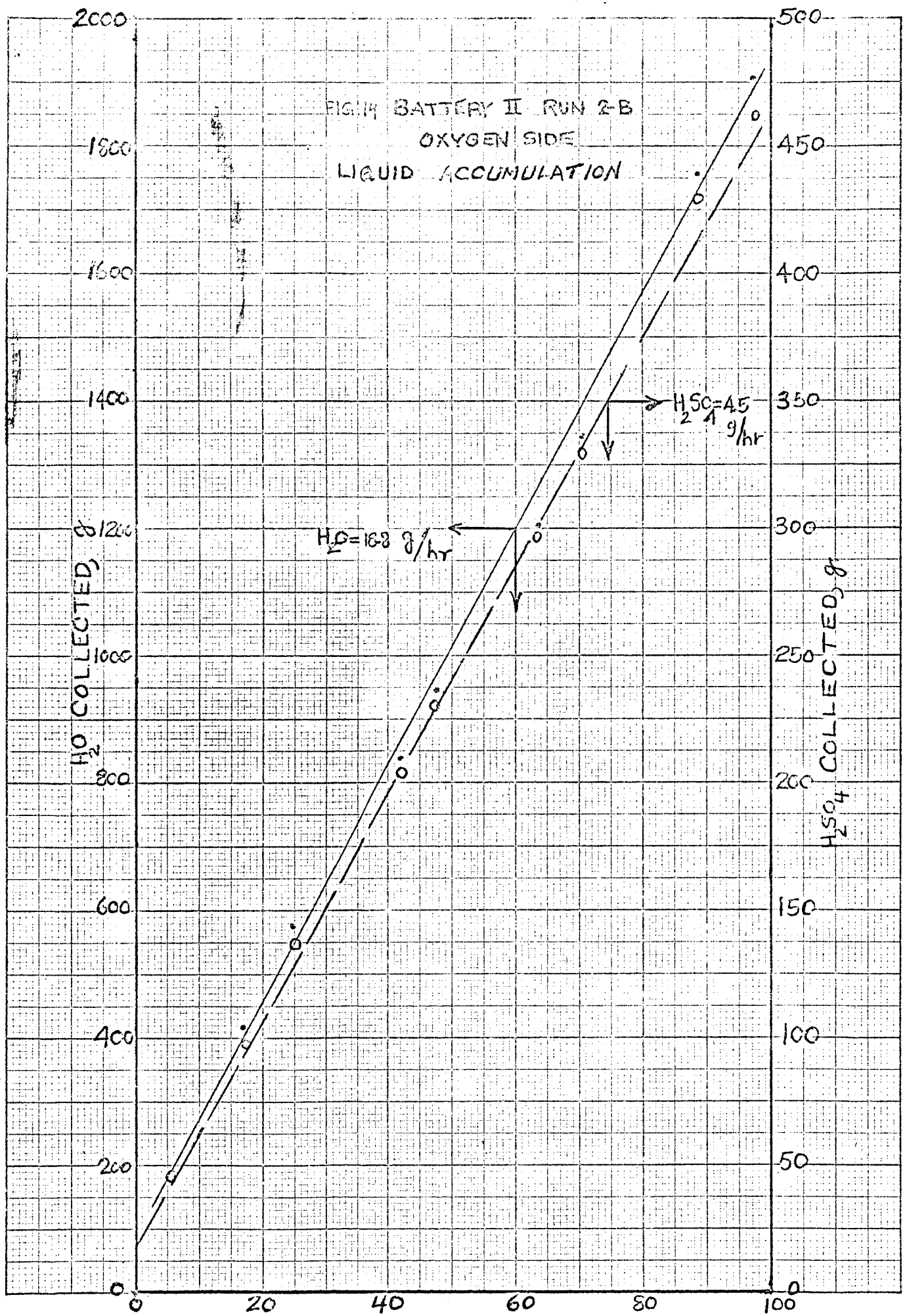
BATTERY 5, RUN 513
TOTAL VOLTAGE

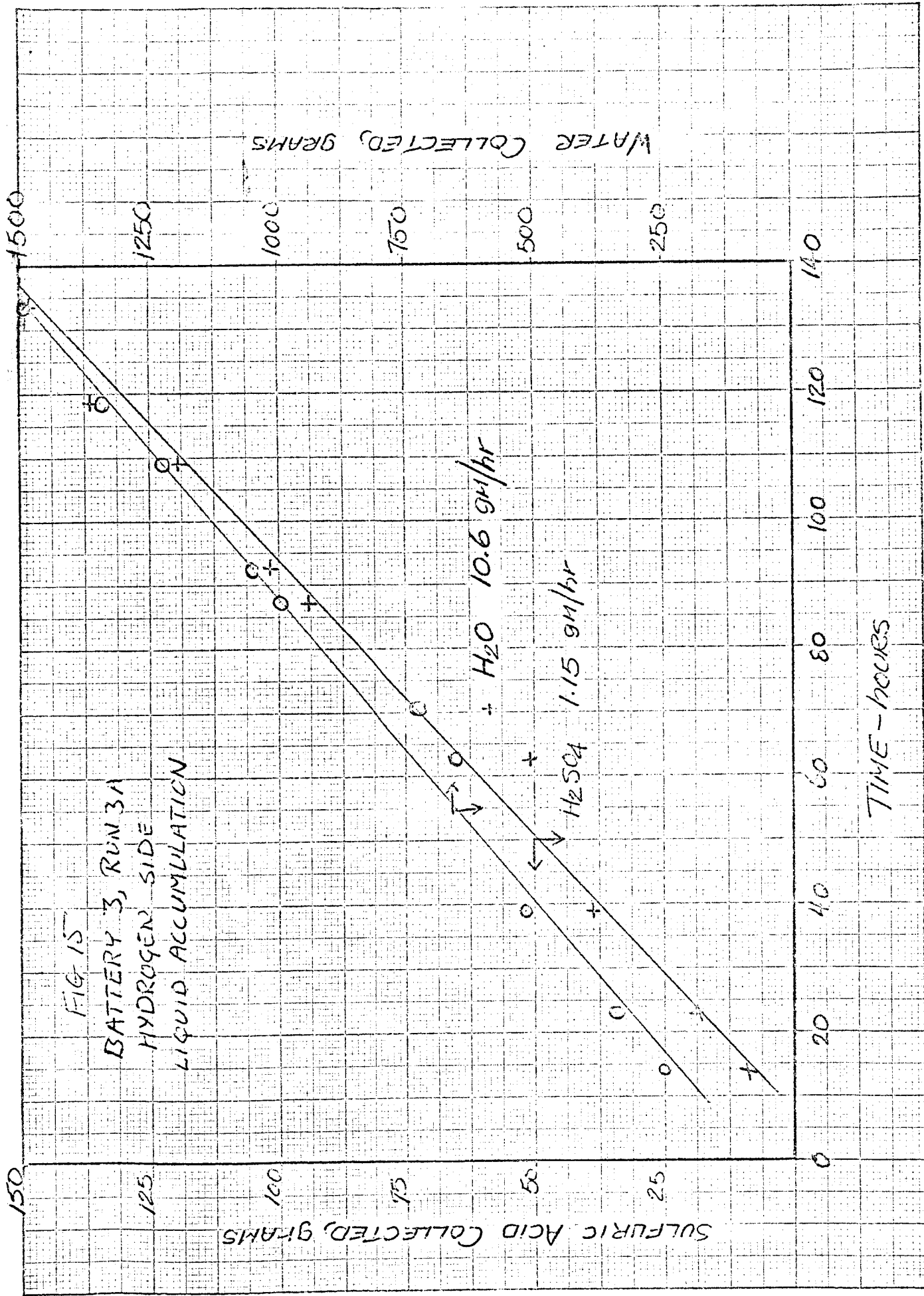


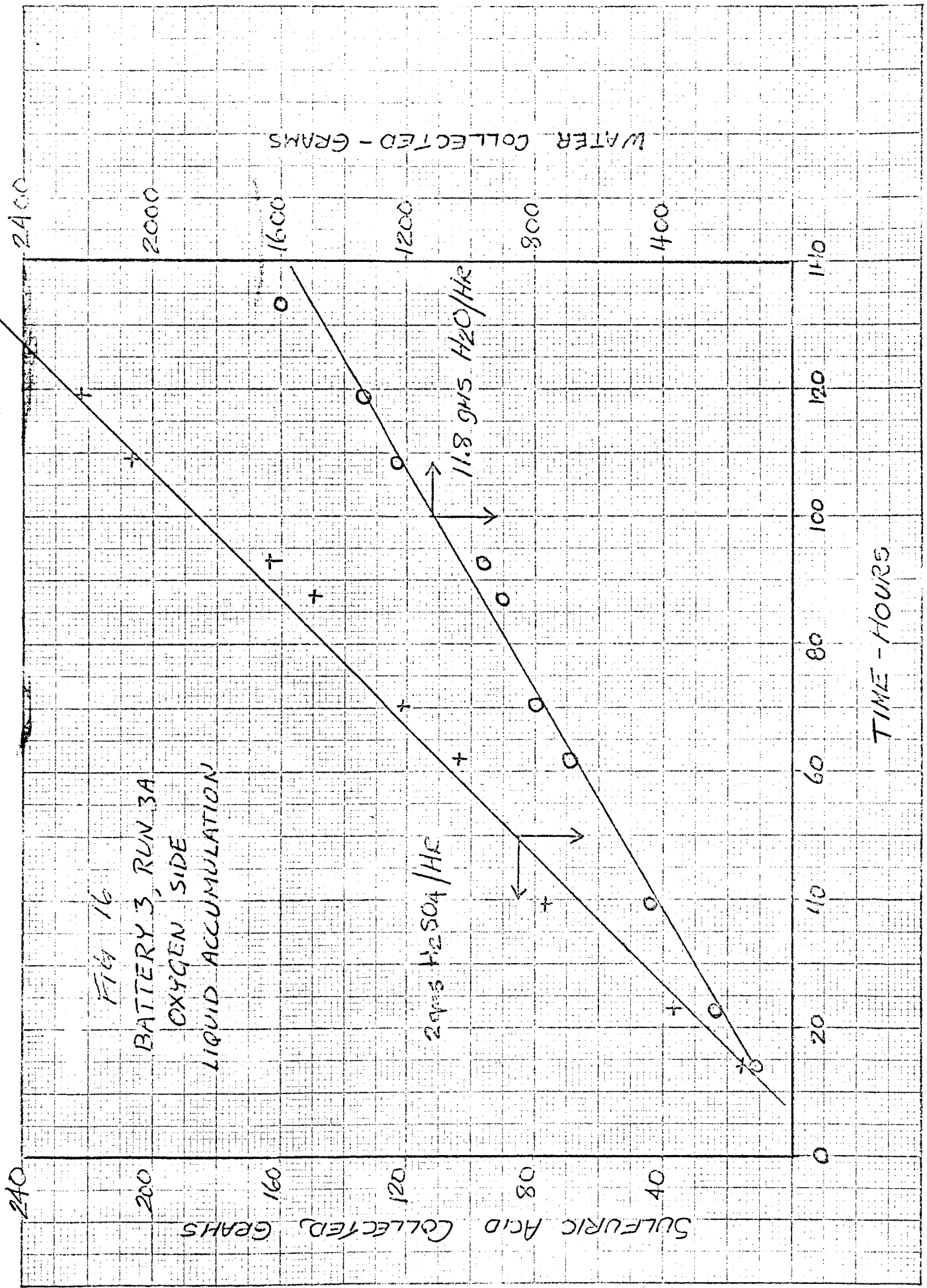
BATTERY 5, RUN 5B
INDIVIDUAL CELL VOLTAGES
24 AMPS / FT²
30°C











150

100

FIG 17

BATTERY 3, RUN 33
HYDROGEN SIDE

LIQUID ACCUMULATION

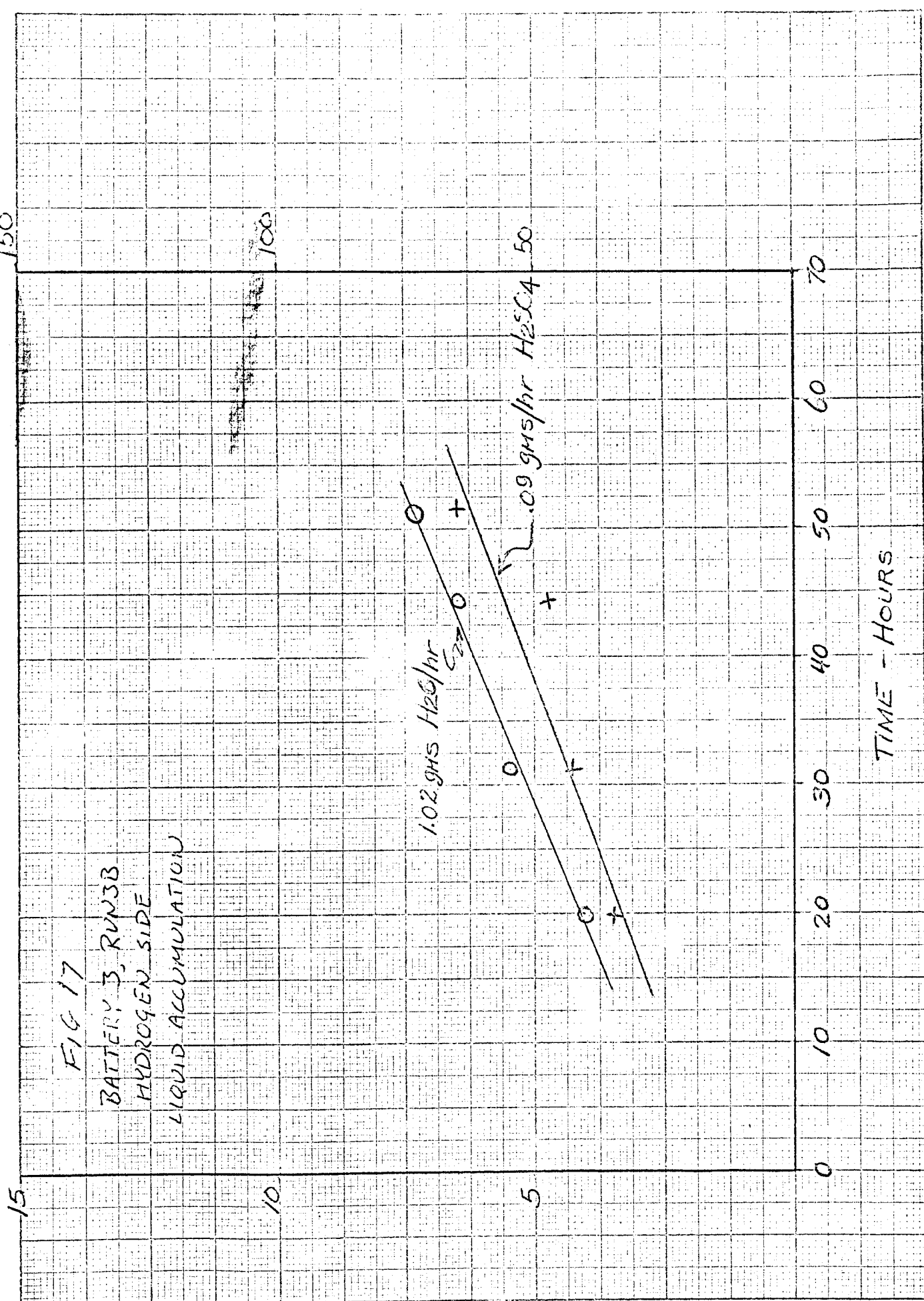
15

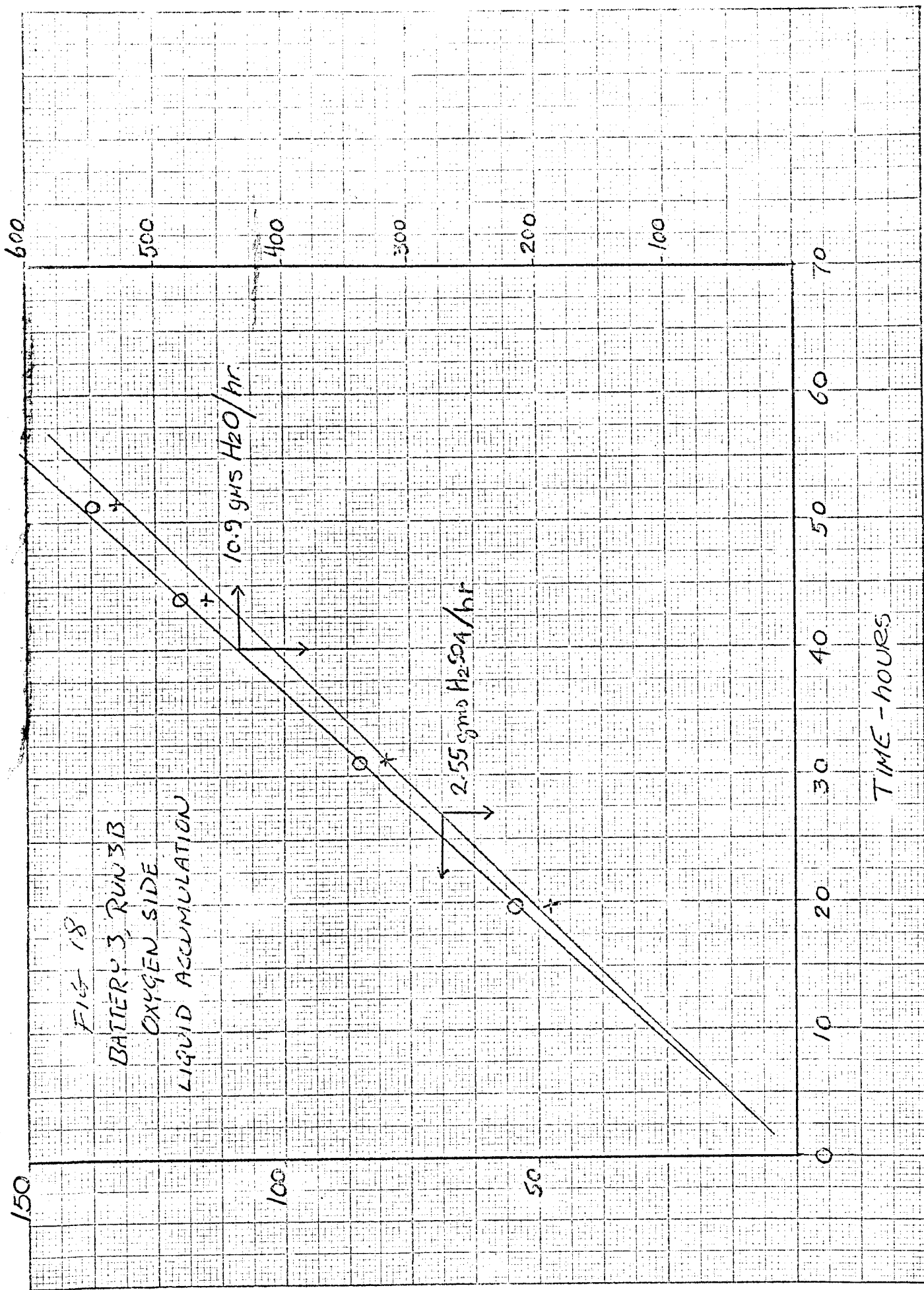
10

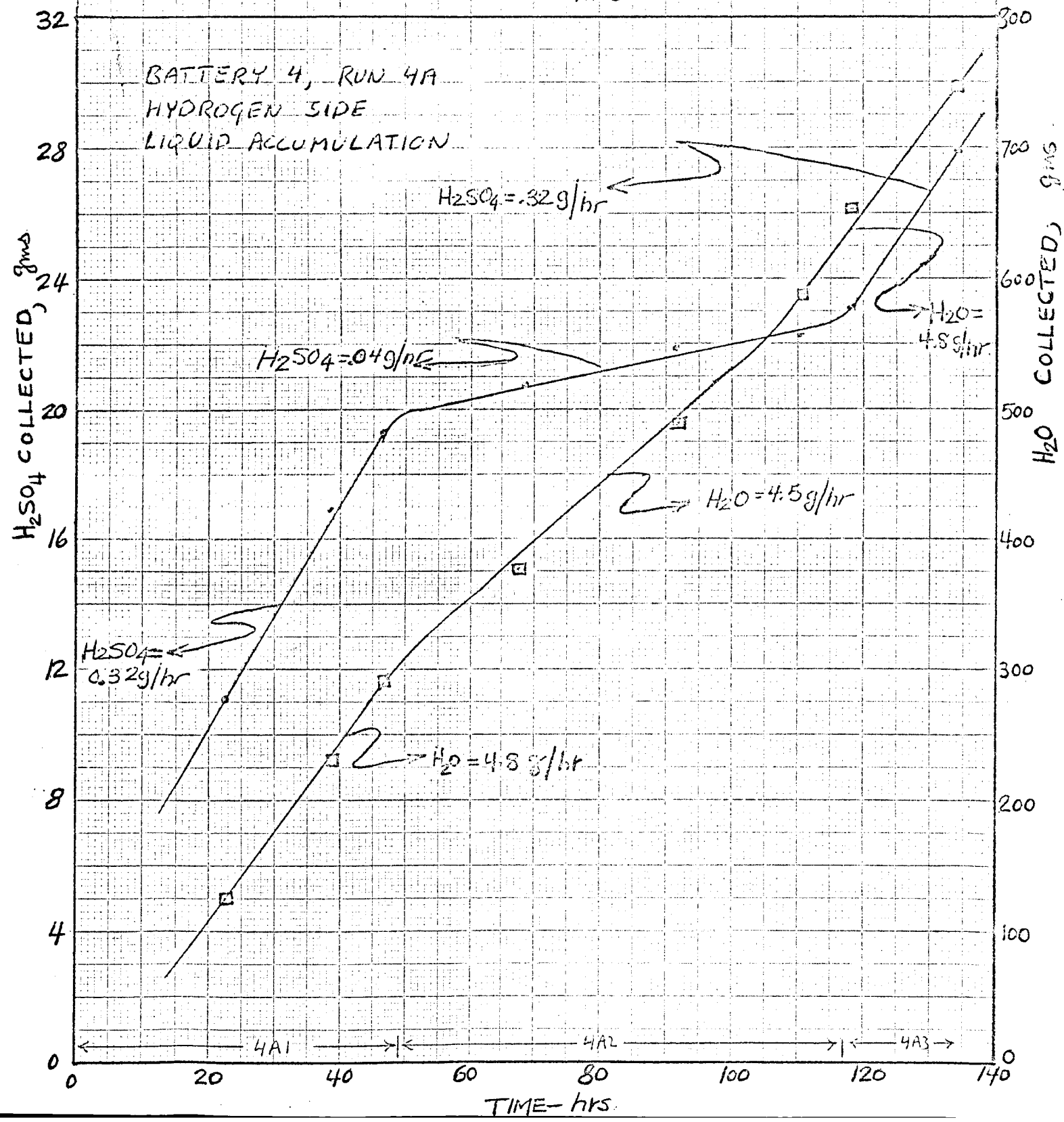
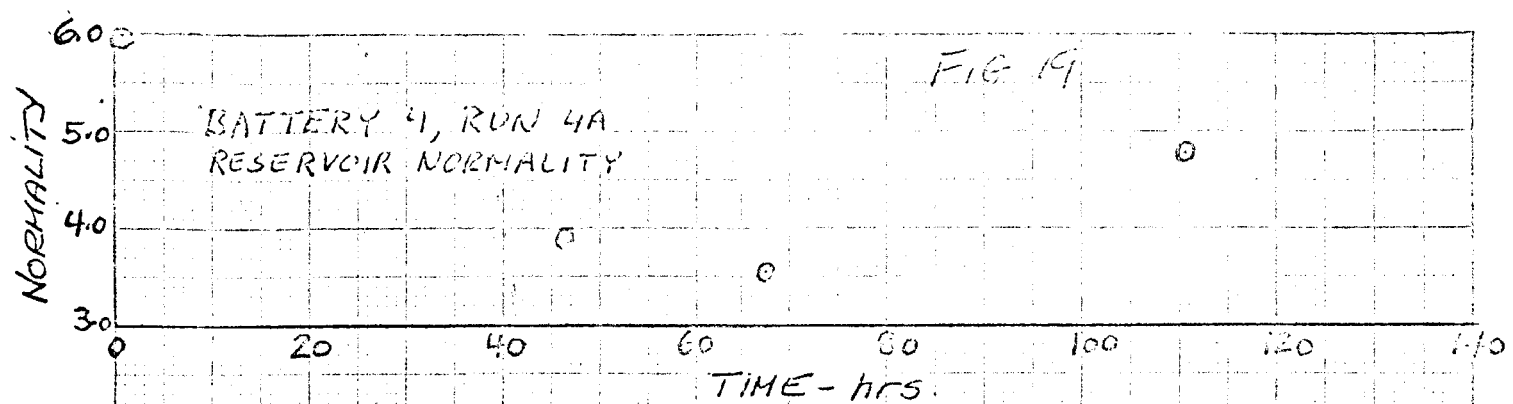
5

1.02 gms H₂O/hr
0.09 gms/hr H₂SO₄

TIME - HOURS







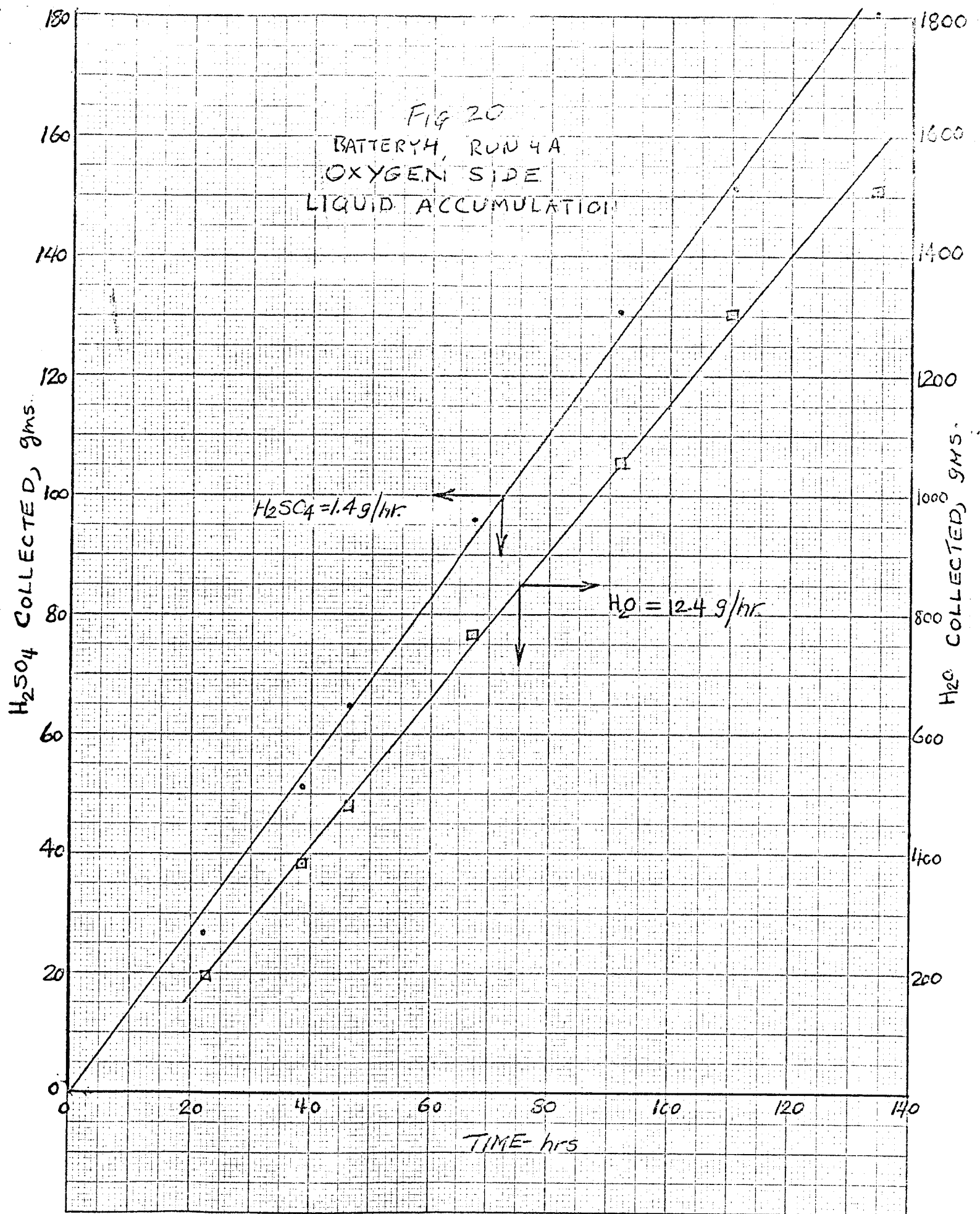


FIG 2-1

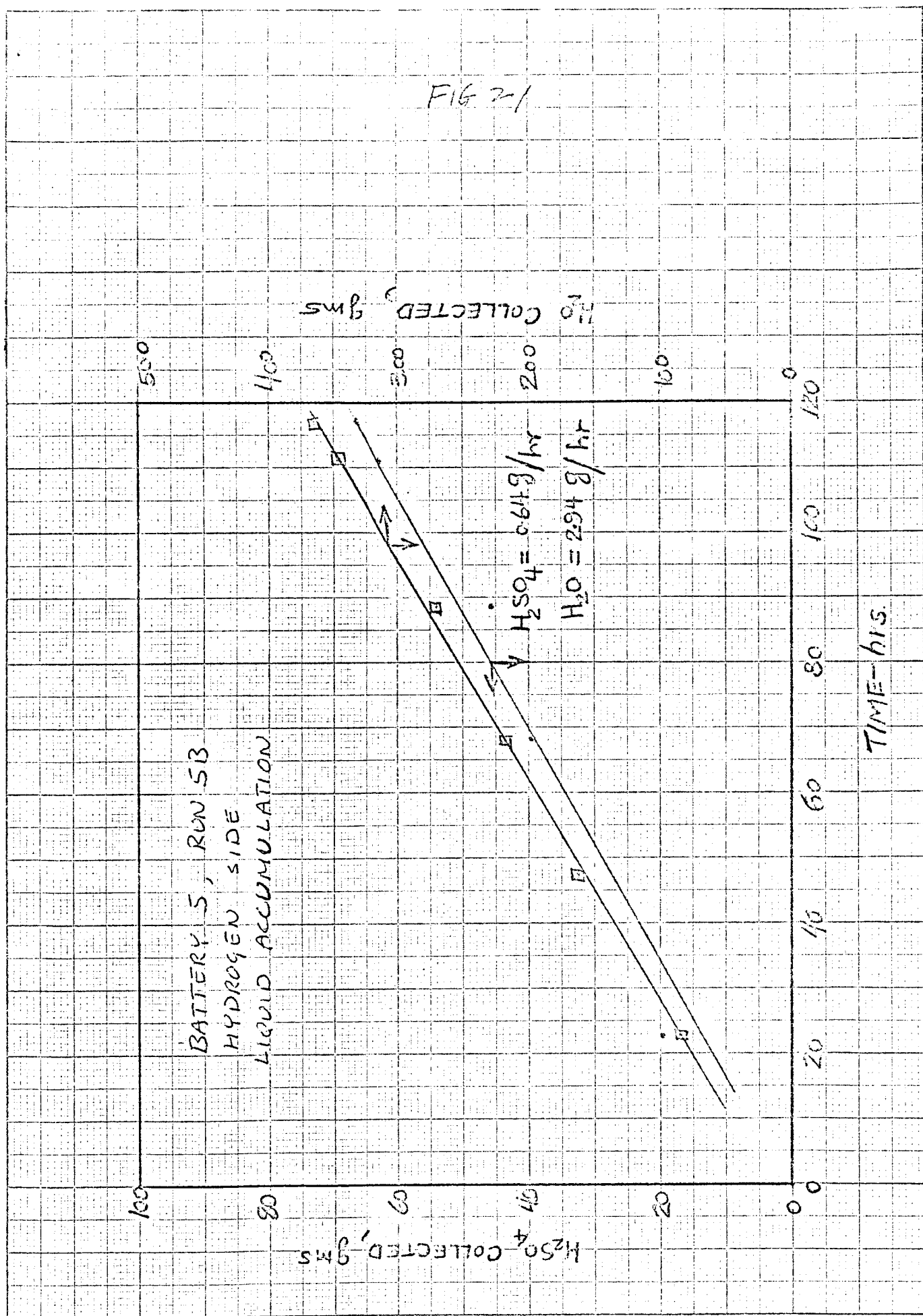


FIG 22

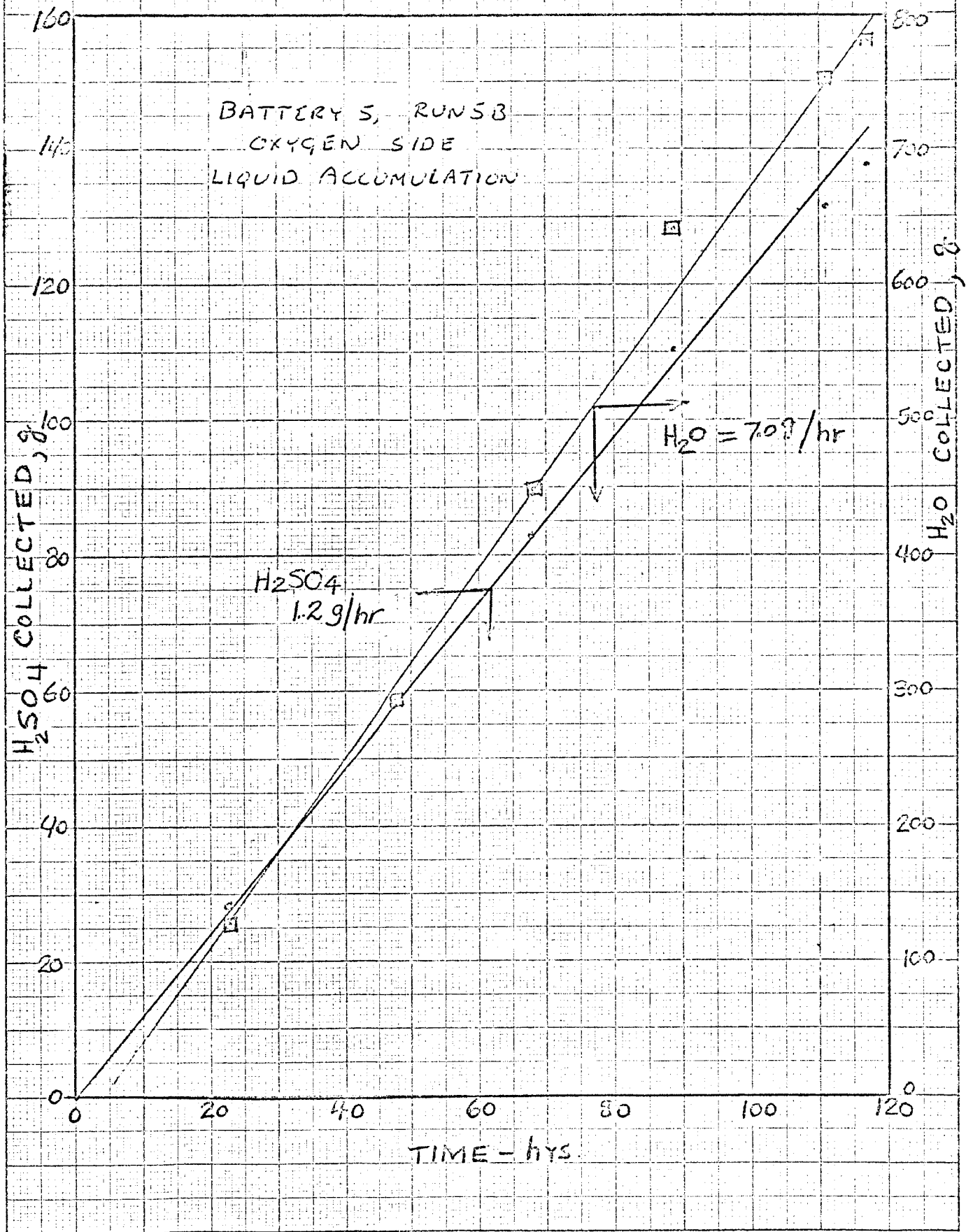


FIG-23

BATTERY 2, RUN 2B
PRESSURE DROPS

H₂ RATE 2.1 LITERS/MIN

O₂ RATE 1.0 LITERS/MIN

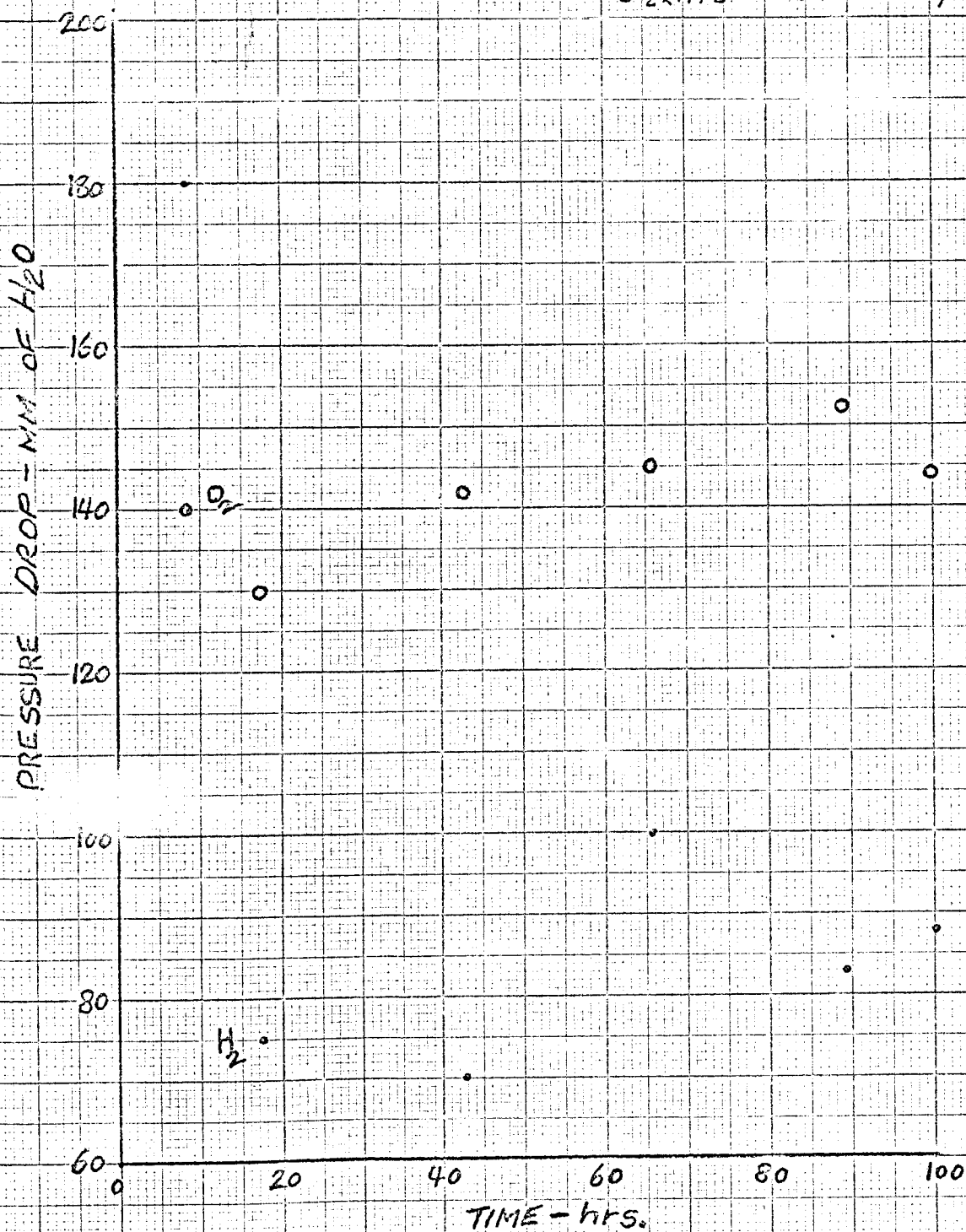


FIG 24

BATTERY 3, RUN 3A
PRESSURE DROPS

H₂ FLOW - 2.1 LITERS/MIN

O₂ FLOW - 1.1 LITERS/MIN

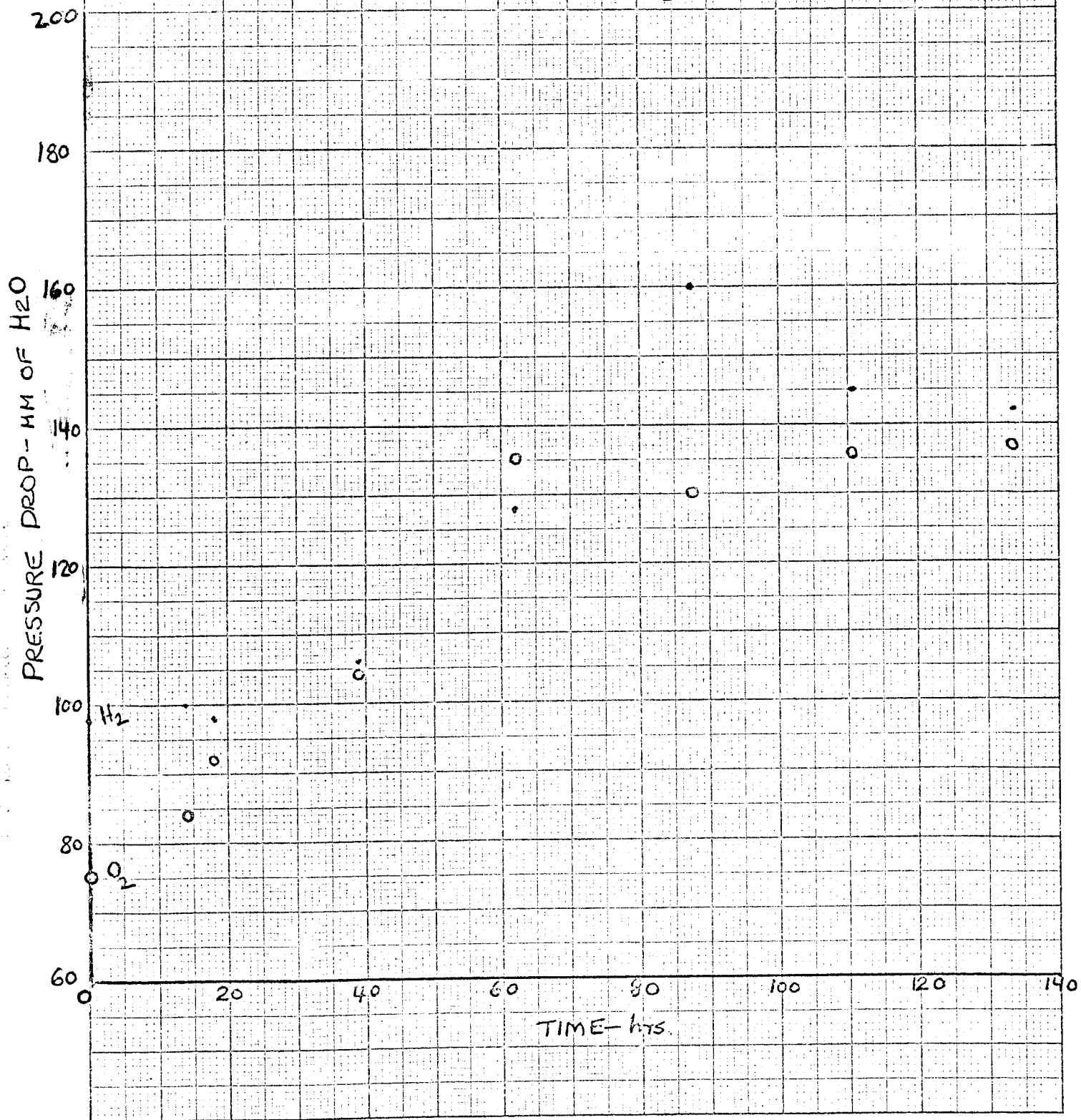


FIG. 25
BATTERY 3, RUN 3B
PRESSURE DROPS
H₂ FLOW - 2.1 LITERS/MIN
O₂ FLOW - 1.1 LITERS/MIN

